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THE EFFECT OF THE MARTENSITIC TRANSFORMATION ON THE
THERMOELECTRIC POWER OF SODIUM

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES IN
PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE
OF MASTER OF SCIENCE

DEPARTMENT OF PHYSICS

by

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EDMONTON, ALBERTA

October, 1960

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ACKNOWLEDGEMENTS

I wish to express my gratitude to Dr. S. B. Woods, my research supervisor, for suggesting this project and for his patient help and encouragement throughout this work.

I am indebted to Mr. P. Crouse who produced the liquid air and liquid helium and provided much technical assistance. Acknowledgements are also due to Mr. J. Legge who made the dewars and was always handy when glassware broke. I wish to thank Mr. J. Rogers for assisting in one of the experiments.

It is a pleasure to acknowledge the support of the National Research Council in providing the cryostat as well as funds for the project.

Finally I wish to express thanks to Dr. J. S. Dugdale for a very informative discussion.

ABSTRACT

This thesis describes experiments performed in order to determine the effect of the martensitic transformation on the thermoelectric power of sodium. The thermoelectric power of two unstrained sodium wires was measured before and after transformation using a cryostat designed by White and Woods (1955). Since strain produced by the transformation prevents complete conversion from body-centered cubic to hexagonal close-packed structure, the measurements on the transformed specimen were carried out on a mixture of the two phases. Two models are proposed with the aid of which the thermoelectric power of hcp sodium can be estimated. The measurements indicate that the hcp phase has a thermoelectric power which is about 10% less than that of pure bcc sodium. A separate experiment was performed which showed that this change in thermoelectric power was indeed due to the structural change produced by the transformation and not to the introduction of additional scattering centers such as dislocations or stacking faults.

It is concluded that the lower thermoelectric power of the hcp phase is due to the fact that the Fermi surface is closer to the Brillouin zone boundary in this phase than it is in the bcc phase.

INTRODUCTION

At low temperatures the crystal structure of sodium changes from body-centered cubic to hexagonal close-packed by a diffusionless transformation. Barrett (1948, 1955, 1956), using an X-ray spectrometer at low temperatures, was the first to observe this transformation and classified it as being of the martensitic type. He found that unstrained sodium partially transforms when it is cooled below 36°K to a hexagonal close-packed structure with stacking faults. The transformation could be induced at temperatures below 51°K by straining. Severe cold work at 5°K transformed about half his sample to the hcp phase. Subsequent reversion to the bcc phase started when the sample was heated to about 70°K and was completed at 110°K, or at a somewhat lower temperature if there had been no cold work. Since then the effects of this transformation on some bulk and transport properties of sodium have been investigated. The effects of the transformation on the specific heat of sodium were measured by Martin (1960); the change in volume produced by the transformation by Basinski and Verdini (1959); the stress-strain relationships, by Hull and Rosenberg (1958) and the electrical resistivities of the two phases of sodium, by Dugdale and Guban (1960). An exploratory experiment by S. B. Woods and J. S. Dugdale indicated

that the martensitic transformation produces a change in the thermoelectric power of sodium. It was felt that this problem warranted further investigation since measured values of the thermoelectric power of sodium obtained by MacDonald and Pearson (1953) differed by almost an order of magnitude from those predicted by the free electron theory. Electrical resistivity and thermal conductivity measurements, on the other hand, agree reasonably with the free electron theory.

Due to the nature of the martensitic transformation it is possible to obtain measurements at a particular temperature of the thermoelectric power of the pure bcc phase as well as the thermoelectric power of a mixture of bcc and hcp phases at the same temperature. This type of measurement is possible anywhere between the onset temperature of the transformation and the subsequent reversion temperature at which the return to bcc structure begins (between T_{α} and T_{γ} , figure 15). The amount of each phase present in the mixture may be determined by simultaneous electrical resistivity measurements using the values for the electrical resistivity of each of the two phases obtained by Dugdale and Guban (1960).

In order to determine the effect of the martensitic transformation on the thermoelectric power of sodium, and to be able to deduce if possible from these measurements the absolute thermoelectric power of the hcp phase, an

apparatus capable of determining accurately temperatures and small temperature differences as well as measuring dc voltages to better than $0.01 \mu\text{v}$ had to be constructed.

This thesis is divided into two parts; the first of these describes the apparatus which consists of a low temperature cryostat designed by White and Woods (1955) and a temperature controller built following the design of Dauphinee and Woods (1955). The temperature controller is capable of temperature regulation within a few thousandths of a degree over a range from 4°K to room temperature. A system capable of measuring dc voltages as low as 5×10^{-9} volts was also built. This system was used to measure the thermoelectric power of the sodium as well as the voltages from the thermocouples with which the temperature gradients in the sodium specimens were detected.

The second part of this thesis deals with the effect of the martensitic transformation on the thermoelectric power of sodium. The experimental procedure and results are discussed. The absolute thermoelectric power of the hcp phase of sodium is deduced and the effect of the structural change produced by the transformation on the shape of the Fermi surface as well as its proximity to the zone boundary are also discussed.

There are two appendices; the first of these deals with measurements of thermoelectric power of lead which

were made in order to obtain absolute values of thermoelectric power. Appendix II shows an extruder which was used in making the lead and the sodium specimens.

PART I

DESCRIPTION OF THE APPARATUS

I THE APPARATUS

1.1 The Cryostat.

The cryostat designed by Drs. S. K. Woods and G. F. White (1955) was kindly provided by the National Research Council of Canada. This type of cryostat is extremely versatile as it enables measurements of electrical resistance, thermal conductivity and thermoelectric power of solids to be made over a range of temperatures from 1°K to room temperature.

The cryostat is shown in the simplified diagram, figure 1. The brass outer can, C_2 , is about 3" in diameter and is suspended by pumping tubes P_1 , P_2 and P_3 which are made of thin walled german silver in order to minimize the heat influx. The inner can, C_1 , is made of copper and plated with gold which has a low emissivity and thus minimizes heat transfer by radiation.[#] Both these cans can readily be removed to allow mounting of different specimens in specimen chamber X. A needle valve (not shown) which may be operated from the top of the cryostat, allows passage of the refrigerant liquid, L, from the dewar into the inner chamber, Q, through tube, V. The inner can, C_1 , may be made vacuum tight and the space between the two cans may then be evacuated through pumping tube, P_3 . The specimen chamber is thus thermally isolated from the refrigerant bath, L, except for the small heat

[#] Copper if highly polished has a lower emissivity than gold but oxidizes too readily.

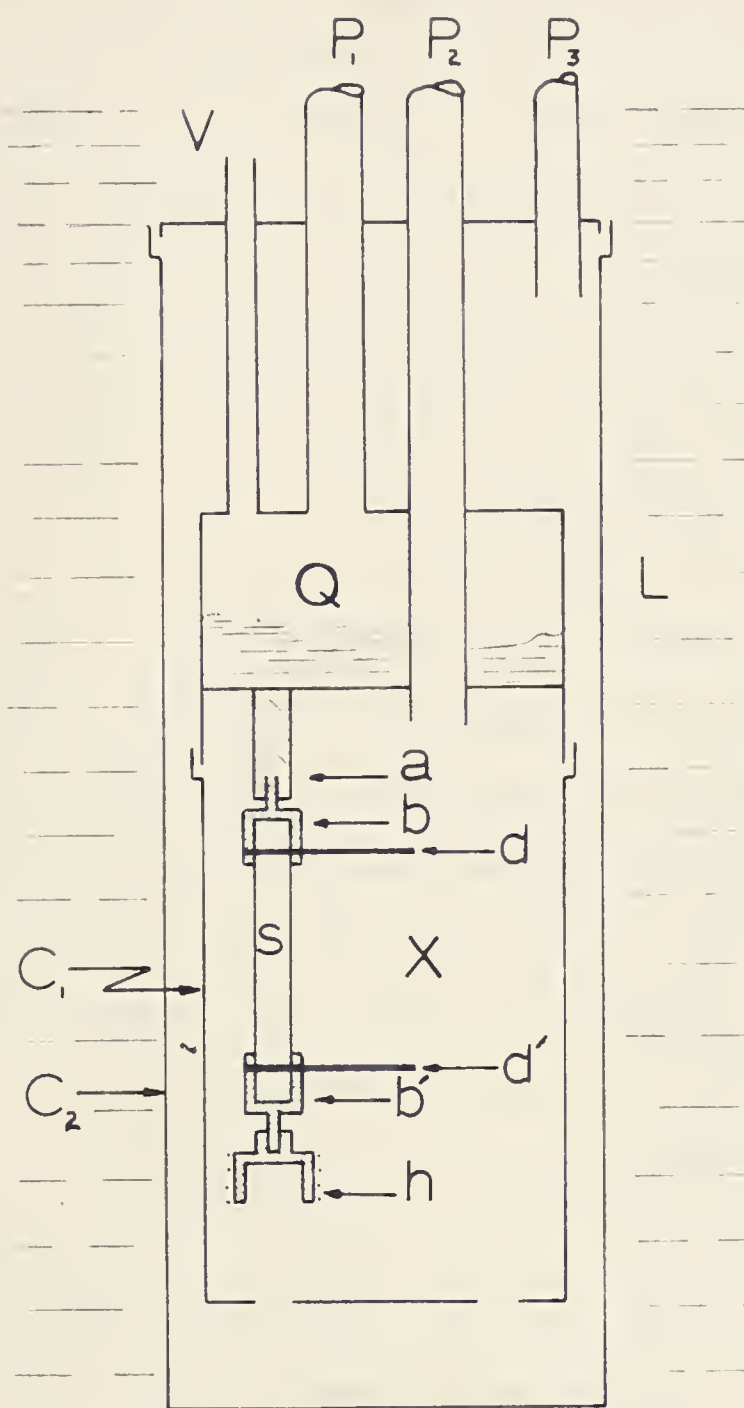


Figure 1. The Cryostat.

leak along the german silver tubes. The specimen chamber may then be cooled below the temperature of the bath by reducing the pressure over the liquid boiling in the inner chamber, Q , by pumping through tube, P_1 . Similarly the specimen chamber can be maintained at temperatures above that of the bath in the dewar by using an electrical heater. A 1000 ohm carbon resistor mounted in a copper sleeve which is soldered on top of the inner chamber serves as the heater. Post, a , specimen holder cap, b and the inner chamber, Q , are all made of copper to facilitate heat transfer to the specimen, S . If the whole specimen is to be kept isothermal (this might be desired, for example, if precision electrical resistance measurements are to be made) then helium exchange gas may be introduced through pumping tube, P_2 . On the other hand, if thermal conductivity or thermoelectric power is to be measured, a temperature difference between the ends of the specimen is required and may be attained using the small electrical heater, h , (described in section 1.43) to heat the bottom end of the specimen while maintaining the other end at a fixed temperature. The specimen chamber must then be kept evacuated by pumping through tube, P_2 , in order that heat conduction take place in the specimen and not elsewhere.

When the experiment involved soft metals, as was the case in the present work (sodium and lead), the specimen was mounted in a specimen holder whose end pieces are made of

copper and are designated as a and b' in figure 1. One inch lengths of copper wire, d and d' , about 0.032" in diameter, are inserted into the specimen through holes in the sides of the end pieces; the potential leads and thermocouples are then attached to the copper wires in such a way that the potential leads make good electrical contact while the thermocouples make good thermal contact but are electrically insulated from the wires. All leads except the potential leads are brought out through pumping tube P_2 . The potential leads, which must be wired to the superconducting reversing switch described in sections 1.4 and 1.41, are brought out through a platinum-glass seal on the top of the outer can. Since the measurement of thermoelectric power is the primary concern in this work, a vacuum is required in both the inner and outer cans so the inner can was not made vacuum tight but had ducts through its wall so that the potential leads could be brought to the outer can and thence to the platinum-glass seal. Both cans could then be evacuated using pumping tube P_2 . The mounting of the specimen and experimental method will be described in Chapter III.

For operation below 55°K two dewars are used; the first of these contains the liquid helium bath which surrounds the cryostat while the second dewar contains the liquid air around the helium dewar. Each dewar is made of pyrex with the surfaces next to the vacuum space

silvered except for two strips about $\frac{1}{8}$ " wide diametrically opposite one another and extending for the length of the dewar. These strips provide windows through which the liquid level can be determined. The liquid air dewar is evacuated and permanently sealed while the liquid helium dewar has a nipple and stopcock in the outside wall near the top so that any helium which has diffused through the pyrex can be pumped out between experiments. For operation above 55°K a single dewar containing liquid air or liquid oxygen is used.

1.2 Temperature Control

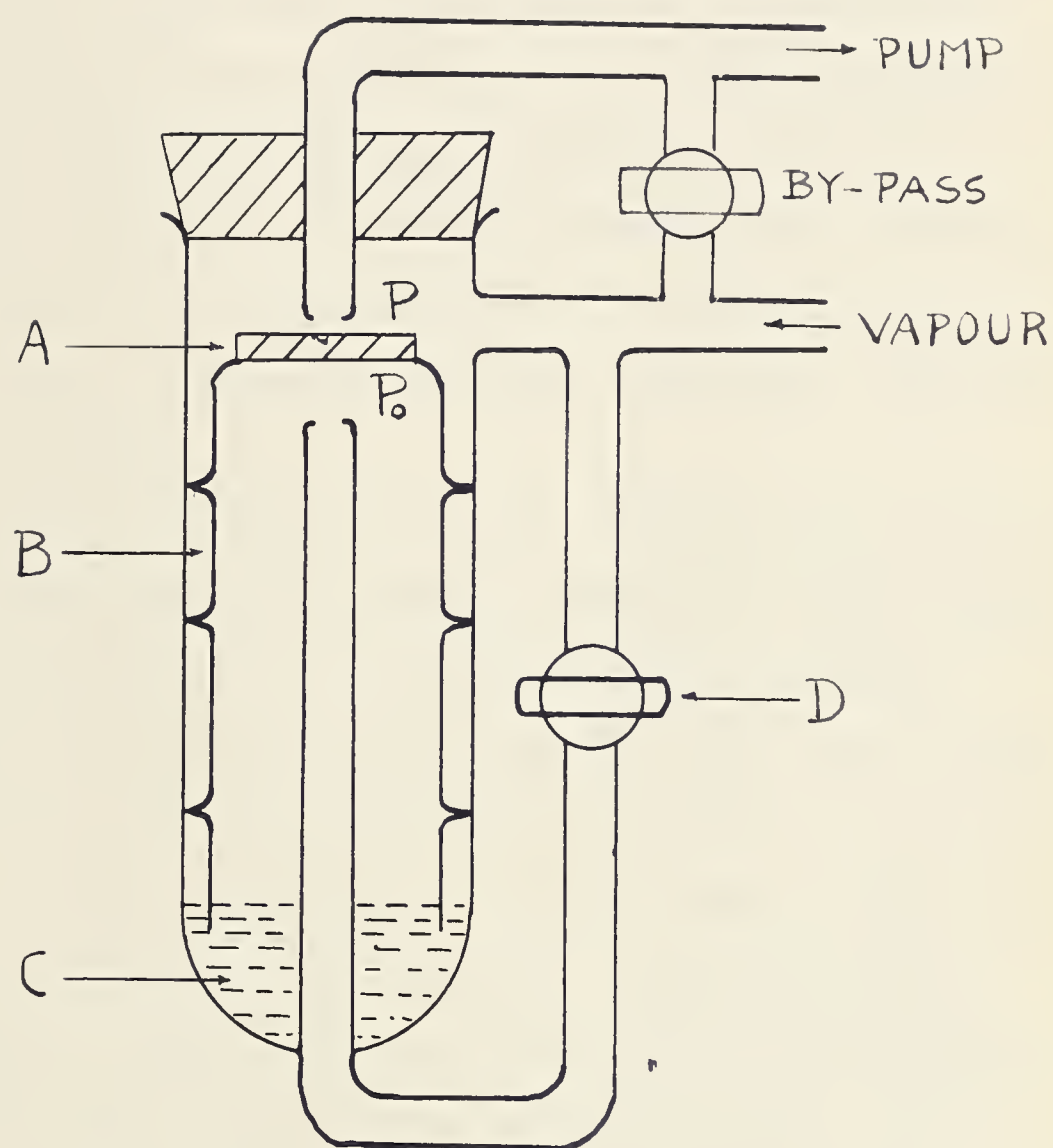
The specimen chamber of the cryostat may be maintained at any temperature between 1 and 300°K. For temperatures below 55°K the cryostat is surrounded by liquid helium boiling at 4°K, while liquid oxygen boiling at 90°K serves as a coolant when the specimen chamber is maintained above 55°K. Temperature regulation is achieved by two separate techniques:

a). Temperatures below that of the coolant bath are controlled by maintaining a constant pressure below one atmosphere over liquid boiling in the inner chamber. Liquid helium is used as the working substance in the temperature range from 1 to 4°K, the range from 55 to 90°K being covered using liquid oxygen.

b). In the temperature regions between 4 and 55°K and above 90°K, which are not attainable with boiling liquid helium or liquid oxygen, the temperature of the specimen chamber is electronically controlled to within 0.001°K of any desired temperature above that of the bath in the surrounding dewar vessel.

1.21 Temperature Control at Temperatures Below the Bath Temperature

Liquid from the refrigerant bath can be introduced into the inner chamber through the needle valve which is operated from the top of the cryostat. The temperature



CARTESIAN MANOSTAT.

Figure 2

of the liquid is lowered by pumping on the inner chamber with the needle valve closed until the pressure corresponding to the desired temperature is reached. This pressure is obtained from the vapour pressure-temperature curve for the particular liquid in use. A manometer connected to the inner chamber is used for determining the vapour pressure when it is between 10 mm Hg and atmospheric pressure. Pressures between 2 and 10 mm Hg are measured with a McLeod gauge. The vapour pressures are converted to temperatures using the values tabulated by Linder (1950).

Once the desired pressure is attained it is controlled by a Cartesian manostat shown in figure 2. This manostat, of a type discussed by R. Gilmont (1951), provides regulation of pressures above 2 mm Hg. The glass diver, B, floats in a pool of mercury, C. A rubber pad, A, is cemented to the top end of the diver. The pressure, P, above the liquid is first reduced by pumping through the by-pass. When the desired pressure, P_0 , is reached valve D and the by-pass valve are both closed. If the pressure drops below P_0 the diver will rise and the rubber pad will close the aperture to the pump, but if the pressure rises above P_0 the diver will drop opening the inner chamber to the pump. Thus the pressure will fluctuate with a small amplitude ΔP about P_0 . This system provides a very precise means of controlling temperature.

1.22 The Electronic Temperature Controller

The electronic temperature control system, shown in the block diagram of figure 3, was built following the design of Dauphinee and Woods (1955). Basically the system compares the voltage of a thermocouple with a dc reference signal and amplifying the difference voltage using a chopper amplifier; the amplified signal is then used to control the power fed to a heater within the cryostat.

The thermocouple that serves as a detector consists of Au+ 2.1 atomic percent Co and Ag+ 0.37 atomic percent Au alloy wires. It has a thermoelectric power of about $3.5 \mu\text{v}/\text{deg}$ at 4°K . The overall sensitivity of the system is about $0.01 \mu\text{v}$, thus temperature regulation within a few thousandths of a degree is possible even at liquid helium temperatures and the regulation improves as the temperature is increased due to an increase in the thermoelectric power of the thermocouple. One junction of the thermocouple, consisting of the two alloy wires, is soldered to the copper top plate of the specimen chamber. The other ends of the two alloy wires are joined to two copper wires and thermally anchored to the surrounding vessel which is at the temperature of the refrigerant liquid in the dewar.

The thermocouples are in series with the neutral

THE ELECTRONIC TEMPERATURE CONTROLLER

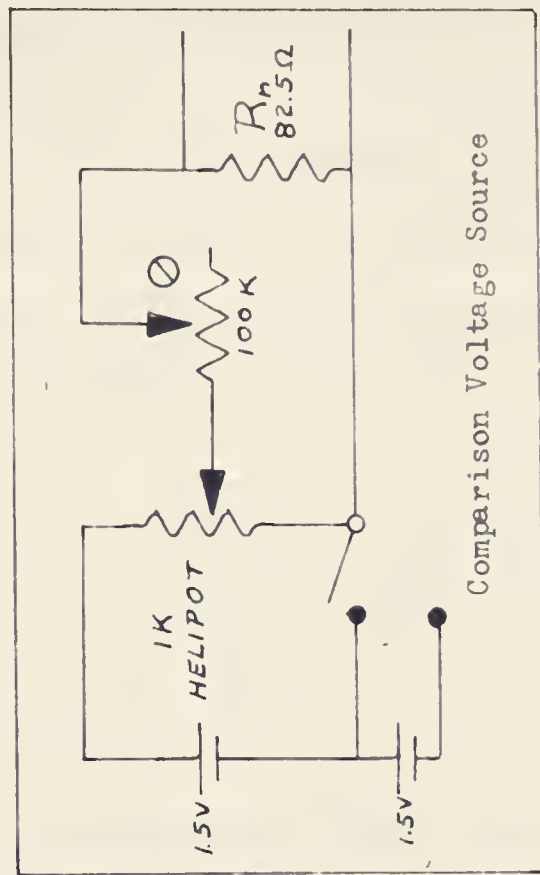
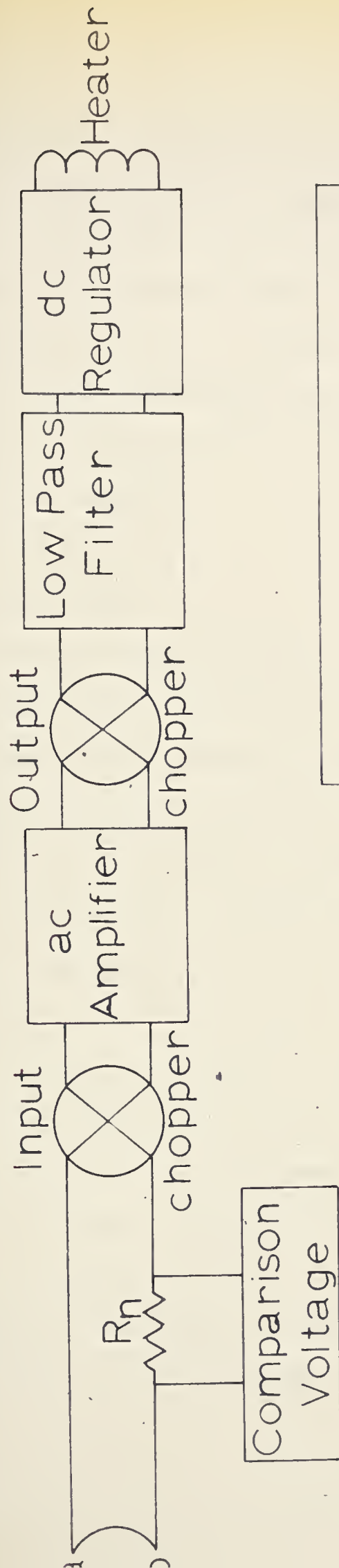


Figure 3

resistor, R_n in figure 3. A comparison voltage which is pre-set so as to be equal in magnitude but of opposite polarity to the thermoelectric force of the thermocouple when the specimen chamber is at the desired temperature appears across the neutral resistor. In this way only the difference between the thermocouple voltage and the comparison voltage appears across the input of the chopper amplifier.

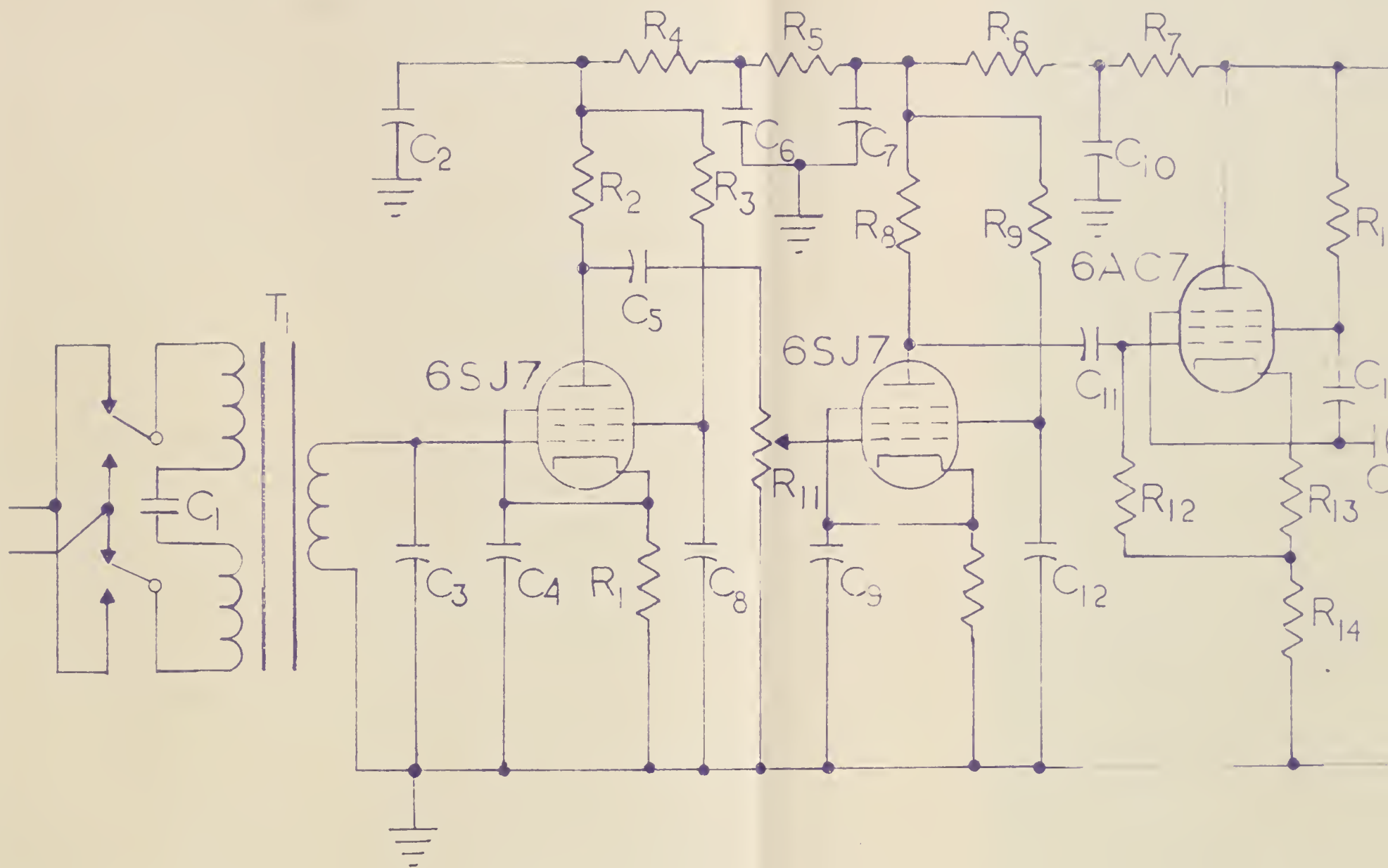
The neutral resistor was made by butt-welding two #38 B&S gauge copper wires to each end of a 26" length of #40 B&S gauge manganin wire. The welds are made by discharging a capacitor through the junction in a manner described by Armstrong and Dauphinee (1947). The manganin wire and several inches of the copper wires were then wound bifilarly on an enameled copper rod 1" long and $\frac{1}{2}$ " in diameter to which they were then cemented. The coil was wrapped in cotton wool and placed in a small box with the four copper leads brought out parallel to one another. A resistor made in this way develops thermal and contact emfs of less than 0.01 μ v even in a room where the temperature is not carefully controlled.

Two leads (current leads) from the neutral resistor were soldered to the comparison voltage source, the remaining two leads (potential leads) were connected in series with the thermocouple. "Low thermal" solder

(70.4% Cd and 29.56% Sn) which has an absolute thermoelectric power of about the same magnitude and sign as that of copper was used in this circuit. The 100K Ω variable resistor, shown in the insert of figure 3, was so adjusted, that the full 1000 scale divisions on the dial of the helipot provide 1 mv across the neutral resistor when a single 1.5 v battery is used. Thus from a knowledge of the thermoelectric force of the thermocouple, it is possible to attain the desired temperature in the specimen chamber by simply setting the appropriate reading on the helipot dial.

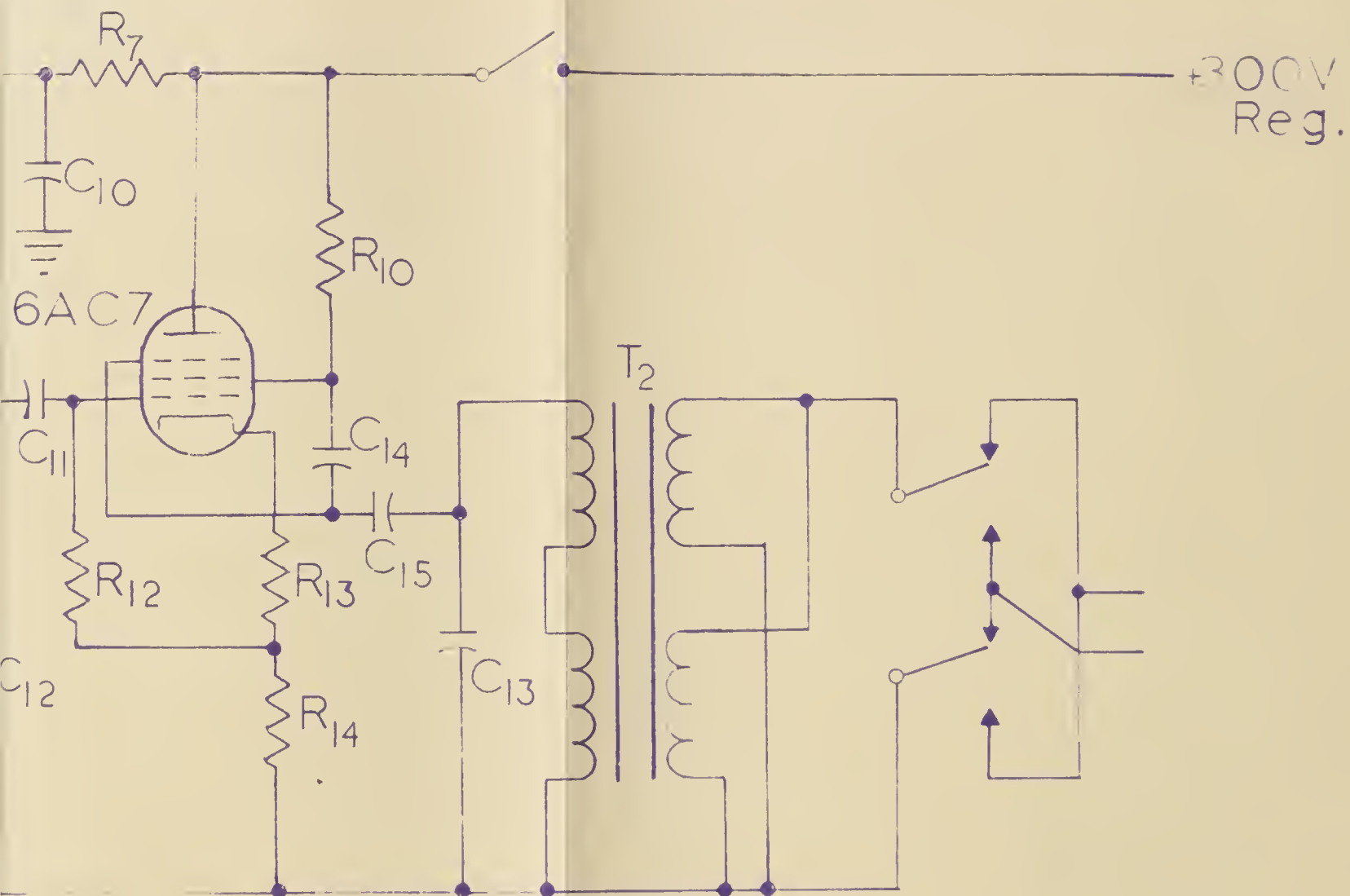
Dc amplification of the difference voltage between the thermocouple and the comparison voltage is accomplished using a chopper amplifier with a maximum voltage gain of about 10^7 . Mechanically driven double pole double throw choppers made by Tinsley of Canada Ltd. (Model TC 112 A) are connected at the input and output of a low audio frequency amplifier which is of conventional design except for the input circuit.

The input circuit of the dc amplifier, shown in figure 4, is rather unique in that it provides for the elimination of thermal voltages arising in the primary circuit of the input transformer as well as contact voltages in the choppers. The thermal voltages are kept



THE DC AMPLIFIER

Figure 4.



PLIFIER

small to begin with by making the input circuit entirely of copper except for the chopper contacts which are made of gold to avoid oxidation of their surfaces. A special input transformer (Hammond 43994) with balanced windings and a low loss mu-metal core is used. A 20 μ f low loss polystyrene capacitor is inserted in series with the primary windings of this input transformer. Any dc signal appearing at the input of the chopper will be converted into a square waveform at the output of the breaker contacts, causing a current to flow through the primary circuit, while any dc voltage arising beyond this point will not experience the action of the choppers and will cause a unidirectional current in the primary of the transformer. Such a current, however, can last only until the capacitor C is charged, after which time no more current will flow. Thus undesired dc voltages in the input circuit are cancelled. The chopper drive is adjusted to provide operation at about 39 cps, the exact frequency being determined by trial and error so that beats with harmonics of the line frequency become too fast for the system to follow.

From the output choppers the demodulated signal passes through a filter stage and a stage of dc amplification to the control grids of a bank of regulator tubes all connected in parallel. A separate 110 volt dc source

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From the output choppers the demodulated signal passes through a filter stage and a stage of dc amplification to the control grids of a bank of regulator tubes all connected in parallel. A separate 110 volt dc source

connected to the plates of these tubes supplies the power for the heater in the specimen chamber, which is connected between the regulator cathodes and ground. A ten turn helipot allows manual adjustment of the heater voltage so that the correct power to maintain the temperature of the specimen chamber may be obtained with zero output from the dc amplifier. The heater itself is an IRC resistor mounted on top of the specimen chamber of the cryostat. This type of resistor is chosen because its resistance is not as temperature sensitive as other makes, such as Allen-Bradley.

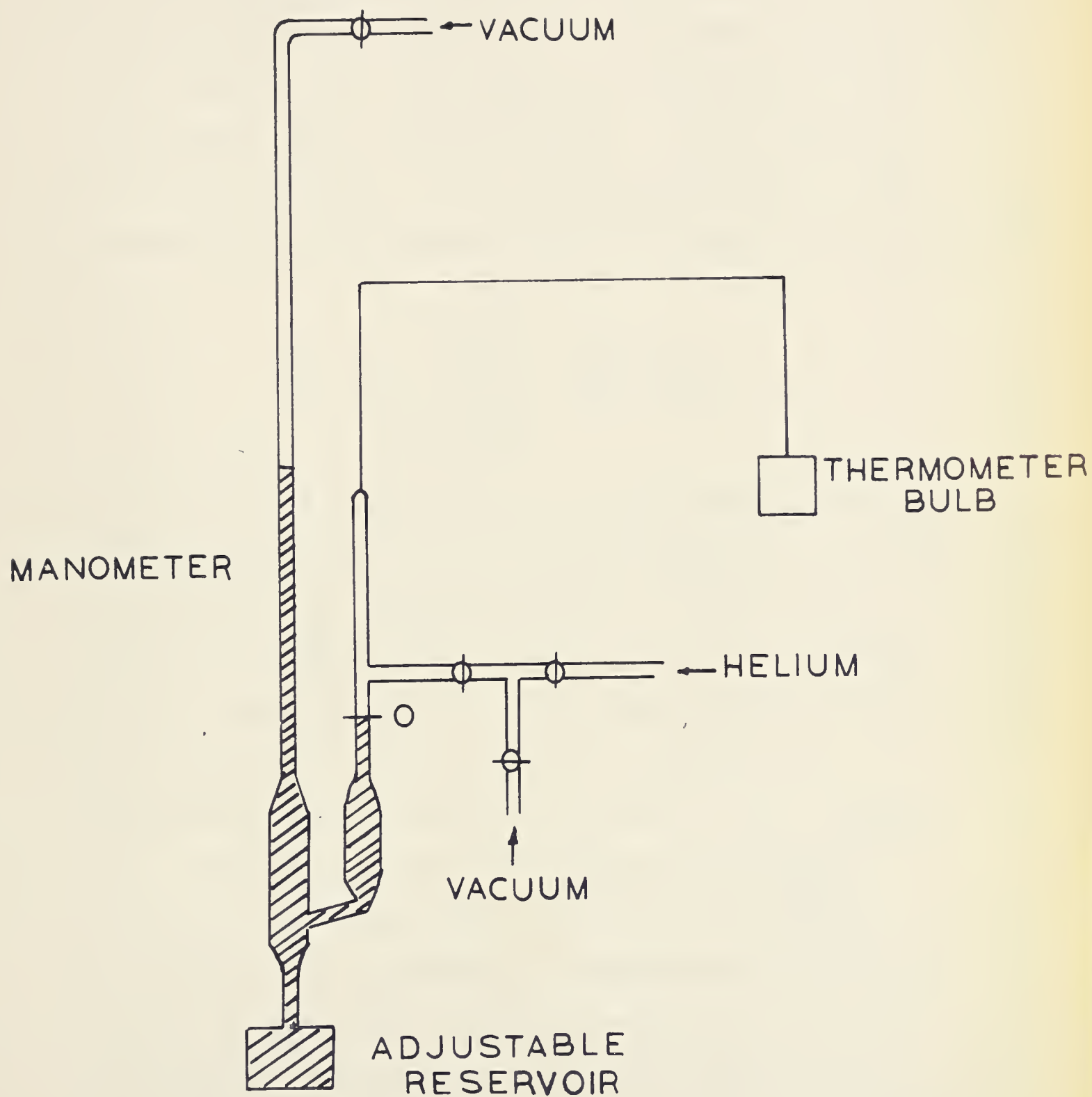
A great deal of care had to be taken with the grounding, shielding and lead dress of the whole system in order to minimize stray ac voltages. The wires connecting the cryostat and controller racks had to be laid out so that they would experience no swaying movements, as such motion induced voltages in the wires large enough to render control impossible. All connecting wires were solid copper, rather than stranded, and were chosen to be as free from strains and kinks as possible in order to reduce thermal voltages. The thermocouple leads were connected to the amplifier by means of solid copper blocks cemented to a heavy brass terminal strip on the cryostat rack to avoid large thermal gradients at these connections.

1.3 Temperature Measurement

Thermocouples were used for measuring the temperature within the specimen chamber. Three different methods of temperature measurement were employed in order to calibrate these thermocouples. They were calibrated directly against a helium gas thermometer, of a type similar to that designed by Sir F. Simon. An indium resistance thermometer was used to measure the temperature at which the gas thermometer was filled. The temperature of the liquid oxygen or liquid air in the dewar which provides a reference for the thermocouples, when the cryostat is operated between 55°K and 90°K, was determined using the indium resistance thermometer. Vapour pressure thermometry, described in section 1.21, provided a check on the gas thermometer readings in the region from 55 to 90°K. Below 4°K the thermocouples were calibrated directly against the vapour pressure of helium.

1.31 Gas Thermometry

The helium gas thermometer is shown schematically in figure 5. The thermometer bulb, which is made of copper, is connected by a german silver capillary to the manometer system. The gas volume is kept constant by setting the meniscus of the manometer liquid (butyl phthalate) to the fiducial mark, O , before each reading is taken. The adjustment of the level of the



GAS THERMOMETER

Figure 5.

meniscus is accomplished by means of a brass bellows which forms the adjustable reservoir.

For our purposes deviations of the equation of state of helium from the ideal gas laws are negligible in the region above 2°K because the pressures used are never more than 90 cm oil (8 cm Hg). If the system is filled to a pressure P_0 at a known temperature T_0 , the pressure P at a temperature T will be given by the equation:#

$$\frac{T}{P} = \frac{T_0}{P_0} \left[\frac{1 + \frac{T_v}{Vt}}{1 + \frac{T_0 v}{V_0 t}} \right] = \frac{T_0}{P_0} \left[\frac{1 + \Delta}{1 + \Delta_0} \right],$$

where t is room temperature, v is that part of the volume of the thermometer that is at room temperature and V is the volume of the thermometer bulb as determined by direct measurement. The volume, v , may be determined from the above equation by filling the thermometer at room temperature and cooling down to liquid air temperature, thus obtaining two values of T/P . Having found v and assuming that room temperature remains constant, a convenient table of values for $\left[\frac{1 + \Delta}{1 + \Delta_0} \right]$ was drawn up for $T_0 = 79^\circ\text{K}$, the temperature of the liquid air bath at which the thermometer was usually filled. The height of the manometer liquid was measured on a reflecting glass scale which could be read to the nearest $\frac{1}{2}\text{mm}$, providing us with temperature measurements accurate to better than 0.1°K for temperatures

For a derivation of this equation see: A. H. Woodcock
Canad. J. Res. 16 A, 133, (1938).

above 55°K . Since the coefficient of expansion of the thermometer bulb below liquid air temperatures is very small, no correction for contraction of the thermometer bulb is necessary in this temperature region although a small correction is applied at higher temperatures.

The manometer tubes were both made of precision bore capillary of 1.5mm diameter, thus no correction for capillarity was necessary. It should be noted that in the preceding formula no correction term appeared for the gas present in the capillary connecting the thermometer bulb to the manometer. This gas is under a temperature gradient making it difficult to calculate a correction for it. The capillary is of very small bore (0.3mm) so that the volume of gas which it contains is small and the correction is negligible except when the thermometer bulb is at temperatures above 20°K and the capillary passes through liquid helium. Corrections to be applied under these conditions have been obtained by Drs. Woods and White at N. R. C. (unpublished), employing the same equipment but using a platinum resistance thermometer to determine the correct temperature of the bulb. Their values for this correction were used in the present work.

1.32 Indium Resistance Thermometer

A suitable thermometer had to be constructed to measure the temperature at which the gas thermometers were filled, and also the temperature of the reference

junction of the thermocouples. Because it would also be generally useful around the laboratory an indium resistance thermometer similar to that developed by White and Woods (1957) and White, Woods and Anglin (1957) was made. Indium was chosen because it presents several advantages over platinum or other metallic elements for thermometers in low temperature work:

(a). Indium has a low Debye temperature hence its resistance is a linear function of temperature down to very low temperatures. By virtue of this low characteristic temperature it is also soft enough to be easily extruded.

(b). Extremely pure indium is available, having a very low residual resistivity, so that its resistance still remains temperature sensitive at low temperatures.

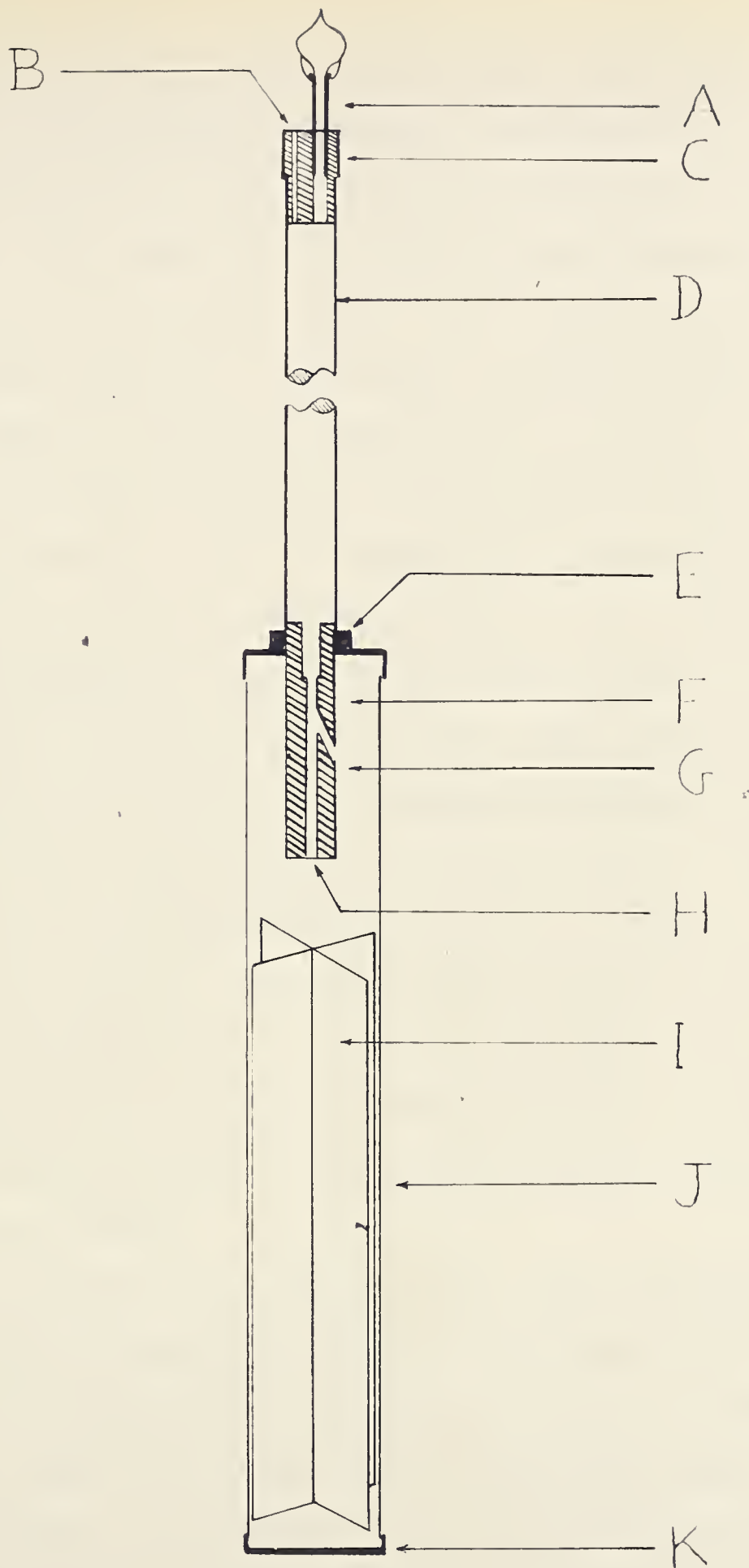
(c). Indium does not go through any phase transformations and hence its resistance remains stable despite thermal cycling.

(d). Indium has a low superconducting transition temperature (3.4°K).

The resistance thermometer was constructed as shown in figure 6. The resistance element of the thermometer was made from a piece of indium of the same purity and from the same source[#] as that used by White and Woods (1958) so their calibration table could be used with confidence.

The indium was extruded into the form of a wire 0.013"

[#] I am very grateful to Mr. G. H. Turner of Consolidated Mining and Smelting Co. of Canada Ltd., Trail B. C., who provided the indium.



RESISTANCE THERMOMETER

Figure 6.

in diameter using an hydraulic press. The indium was wound bifilarly on a coil form, I, made from two sheets of lucite in the form of a cross 0.73" wide and $3\frac{1}{2}$ " in length. This type of coil form provides suspension of the wire with very little strain. The coil form was notched to prevent any of the turns from touching adjacent ones. A $\frac{1}{8}$ " piece of indium was cold welded to each end of the coil to form "Y" junctions. These junctions were in turn indium soldered to four #28 B&S gauge copper wires which served as current and potential leads. The "bulb", J, of the thermometer was made of thin wall inconel tubing, with brass caps E and K soldered to the ends with Wood's metal. The leads of the coil were thermally anchored around the copper form F, and brought up through hole G. Stem D was made of $\frac{1}{4}$ " inconel tubing about 25" long and soft soldered to cap E. The leads were brought out through B, the hole being sealed with de Khotinsky cement. The thermometer was out-gassed through the Kovar glass seal, A, for several hours. It was subsequently filled with helium and sealed.

The circuit used for measuring the resistance of the thermometer is shown in figure 7. To test the reliability of the thermometer it was cooled to liquid air temperature several times and its ice point resistance measured after each cooling. Each time a value of 2.692 ohms was obtained. It can be seen from the diagram of the measuring circuit that the resistance R_T of the indium coil at any

temperature T is given in terms of the voltage V_S across the Kanthal wire resistor and the voltage V_T across the thermometer by:

$$R_T = 1.377 \frac{V_T}{V_S}$$

The residual resistance ratio, R_{42}/R_{273} , for the indium was measured by Messrs. Enns and Quon in this laboratory and found to be 2.6×10^{-4} which agrees well with the value quoted by White and Woods (1957). The thermometer was also checked against pure oxygen boiling at atmospheric pressure and found to agree within 0.2°K with the temperature determined from the vapour pressure measurement.

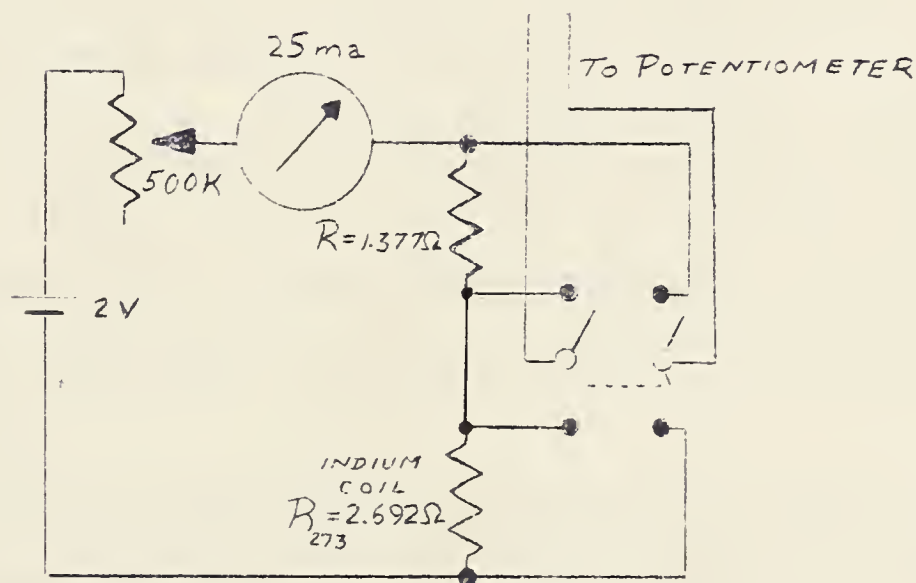
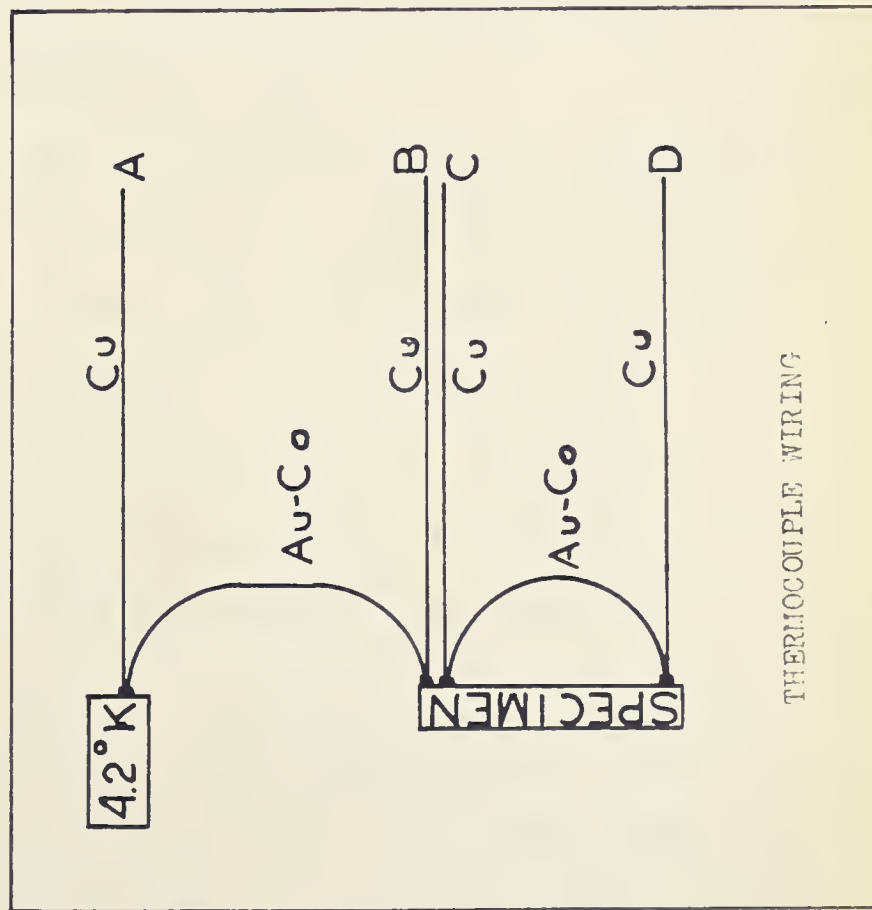
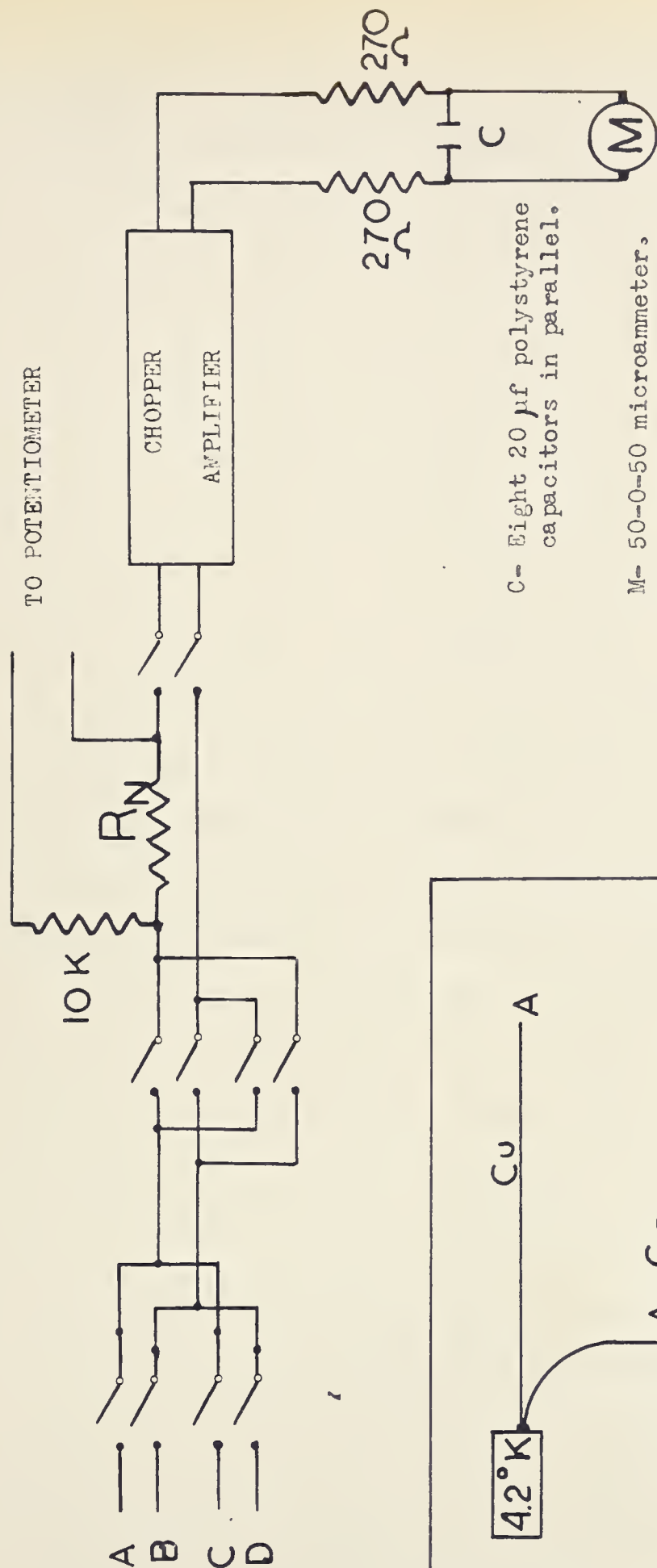


Figure 7. Indium Resistance Thermometer Circuit

1.33 The Thermocouple Measuring System

The thermocouple measuring system consists of two Au + 2.11 percent Co:Cu thermocouples, one of which measures the temperature at the top end of the specimen, while the other measures the temperature difference between two points on the specimen. The thermocouple voltage is compared with a known voltage (supplied by a potentiometer) and the resulting difference signal is amplified using a chopper amplifier similar to that used in the temperature controller described in section 1.22. The only differences between this amplifier and that used in the temperature controller are that in this amplifier a different output transformer is used to provide impedance matching to the output meter and the heater circuit is supplied from a 6 volt storage battery in order to further reduce ac pickup. The output is passed through a filter circuit having a large enough time constant to eliminate undesirable meter flutter, to a center zero microammeter which serves as balance indicator. The whole system is shown schematically in figure 8.

The known comparison voltage appears across R_n , which is a neutral resistor made in the same way as the one used in the temperature controller. This resistor and a 10,000 ohm resistor in series with it are connected across the EMF terminals of a Cambridge Vernier Potentiometer which is the source of the comparison



THERMOCOUPLE SYSTEM

Figure 3.

voltage. The resistance of R_n is such that the voltage appearing across it is $(9.211 \pm 0.05) \times 10^{-3}$ of the potentiometer voltage. The potentiometer is adapted for use as a voltage source by shorting its galvanometer terminals, and a setting of μv on the potentiometer thus corresponds to a voltage of 9.211×10^{-9} volts across the neutral resistor; a signal of about this magnitude is found to give a deflection of about 2mm on the microammeter scale with the amplifier operating at about half gain. Thus thermocouple voltages of the order of a hundredth of a microvolt are easily measured. The comparison voltage can be adjusted in 10^{-8} volt steps so that the amplifier is always used as a null detector and it is not necessary to stabilize its gain particularly or to calibrate the microammeter scale.

Switching between the two thermocouples is achieved by a set of all copper "low thermal" switches. Provision is also made for reversing the polarity of the comparison voltage using the same switches. The thermocouple amplifier was at first mounted on the same rack as the temperature controller but it was found that the interaction between the two chopper amplifiers made such operation impossible. The thermocouple amplifier and potentiometer were then mounted on a separate rack and provided very satisfactory operation after the proper lead dress and grounding arrangements were found. Connecting wires

between the amplifier and the cryostat racks were joined through a terminal strip (with copper connectors) on the cryostat rack. With these precautions the stray voltage in the circuit was less than 10^{-8} volts.

1.34 Thermocouple Calibration

During the calibration runs one thermo-junction was cemented in good thermal contact with the gas thermometer bulb by means of fingernail varnish, while the reference junction was cemented to a copper post which extends through the cryostat wall into the refrigerant liquid. Electrical insulation was ensured with a layer of cigarette paper embedded in the varnish. The thermocouples were calibrated against the gas thermometer between 4 and 90°K . The indium resistance thermometer was used to determine the temperature of liquid oxygen, which was used as the reference bath for all calibrations above 55°K . Vapour pressure measurements on the liquid oxygen in the inner chamber, although they could not be treated with great confidence because of slight impurities in the oxygen, provided a check on the gas thermometer temperatures in this range. Below 55°K the reference bath was liquid helium, the temperature of which was determined from vapour pressure measurements. Below 4°K the vapour pressure of liquid helium in the inner chamber was measured and the thermocouples were

calibrated with temperatures determined from these measurements. Impurities have such low vapour pressures at these temperatures that their effect on the readings may be neglected.

1.35 Discussion of Thermocouple Thermometry

The calibration curves are shown in figures 9, 10 and 11. The values obtained from several runs are consistent within better than 1%. Comparison of this calibration with those obtained by Drs. S. B. Woods and M. T. Elford at N. R. C. (unpublished) and by Bunch, Powell and Corruccini at N. B. S. for similar thermocouples showed good correlation.

It was found that this system provides an extremely convenient method of temperature measurement, since thermocouples are very compact compared with other types of thermometers. Also thermocouples, because of their very low heat capacity react far more quickly to variations in temperature than do gas thermometers or resistance thermometers. Care must be taken however that there are no unnecessary junctions between the thermocouples and the measuring system. Any necessary junction must not be subjected to temperature gradients, since they would then produce thermoelectric voltages of their own. Also kinks and strains in the wires must be avoided as they produce similar effects. Thermoelements

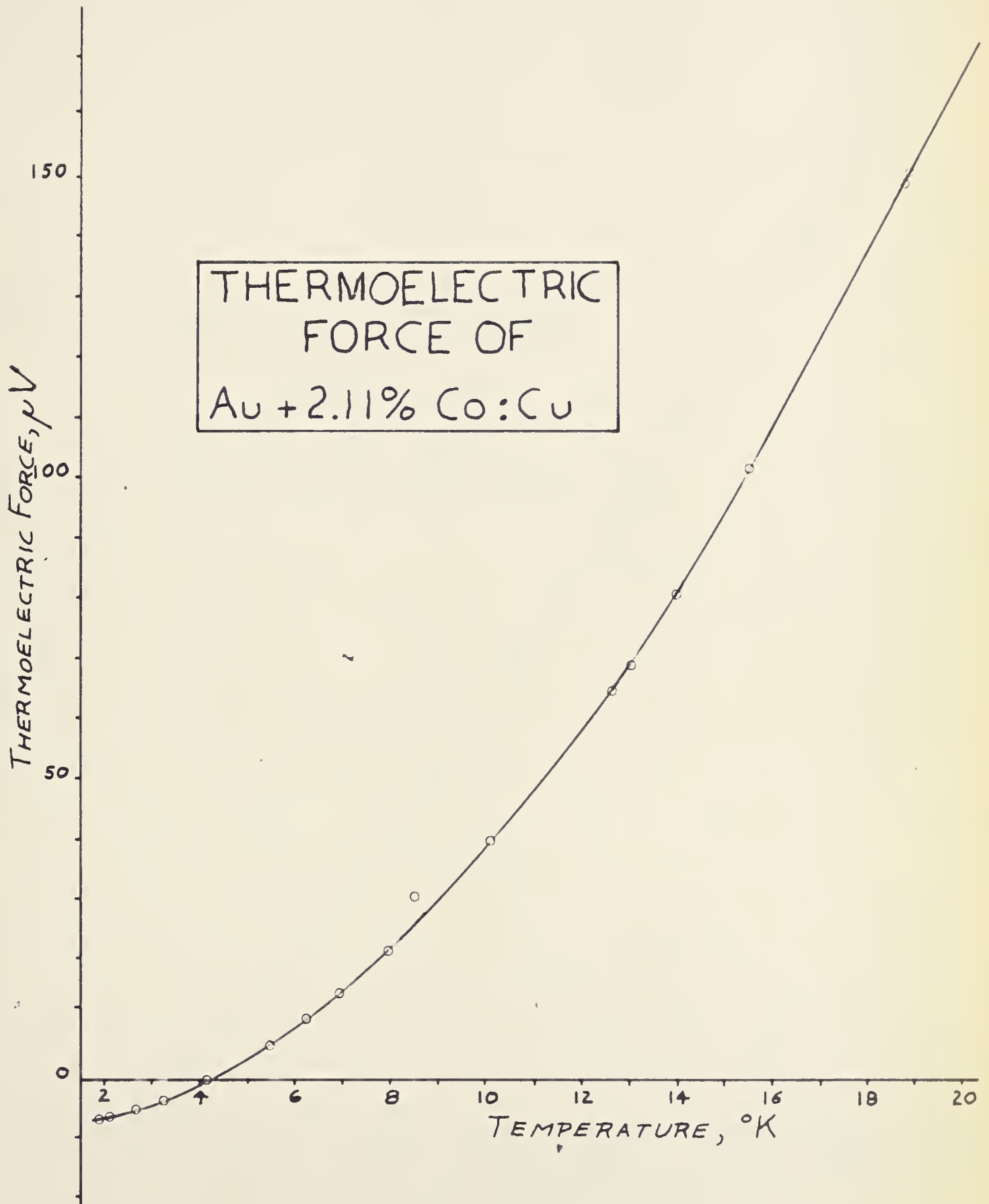


Figure 9.

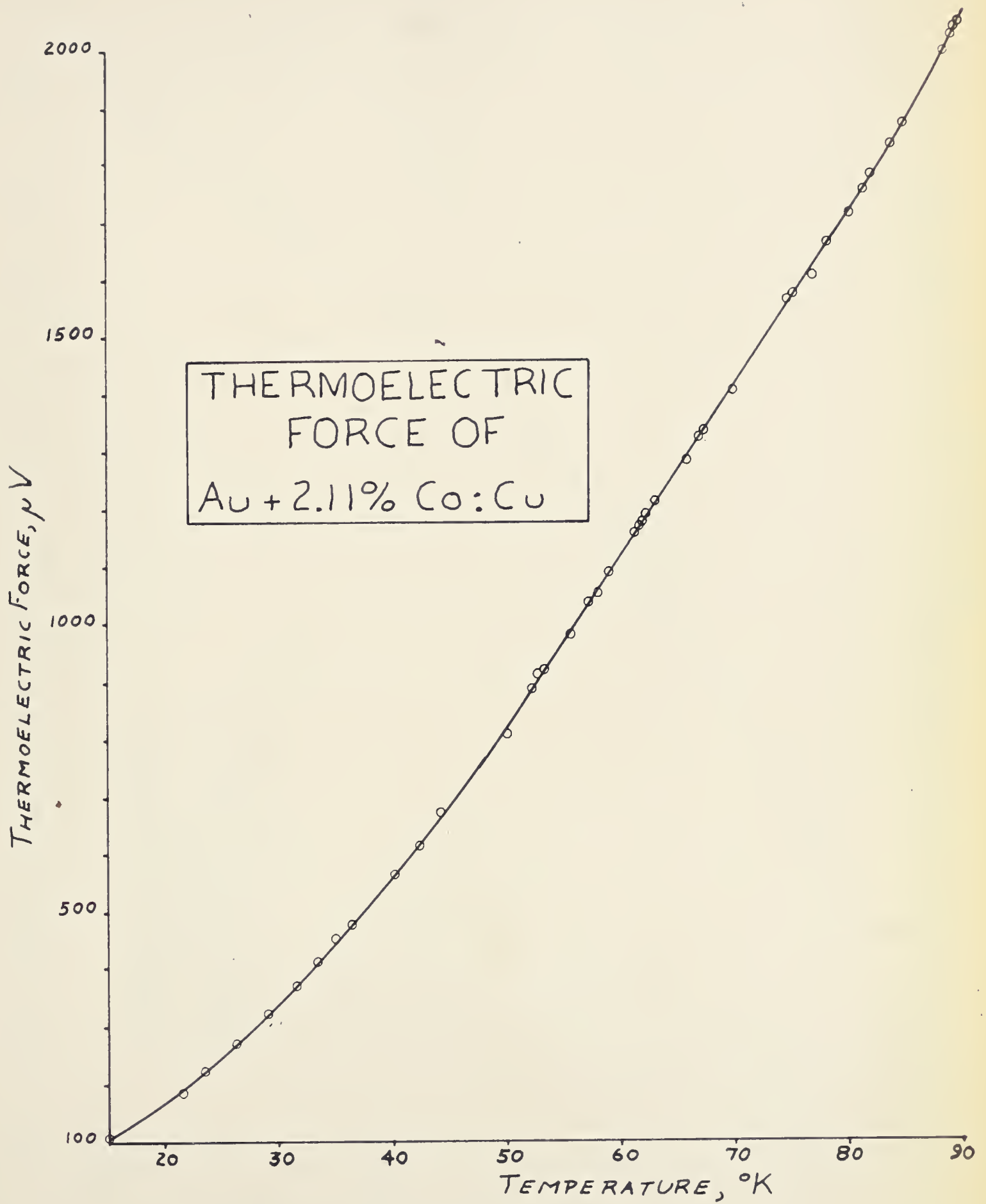


Figure 10.

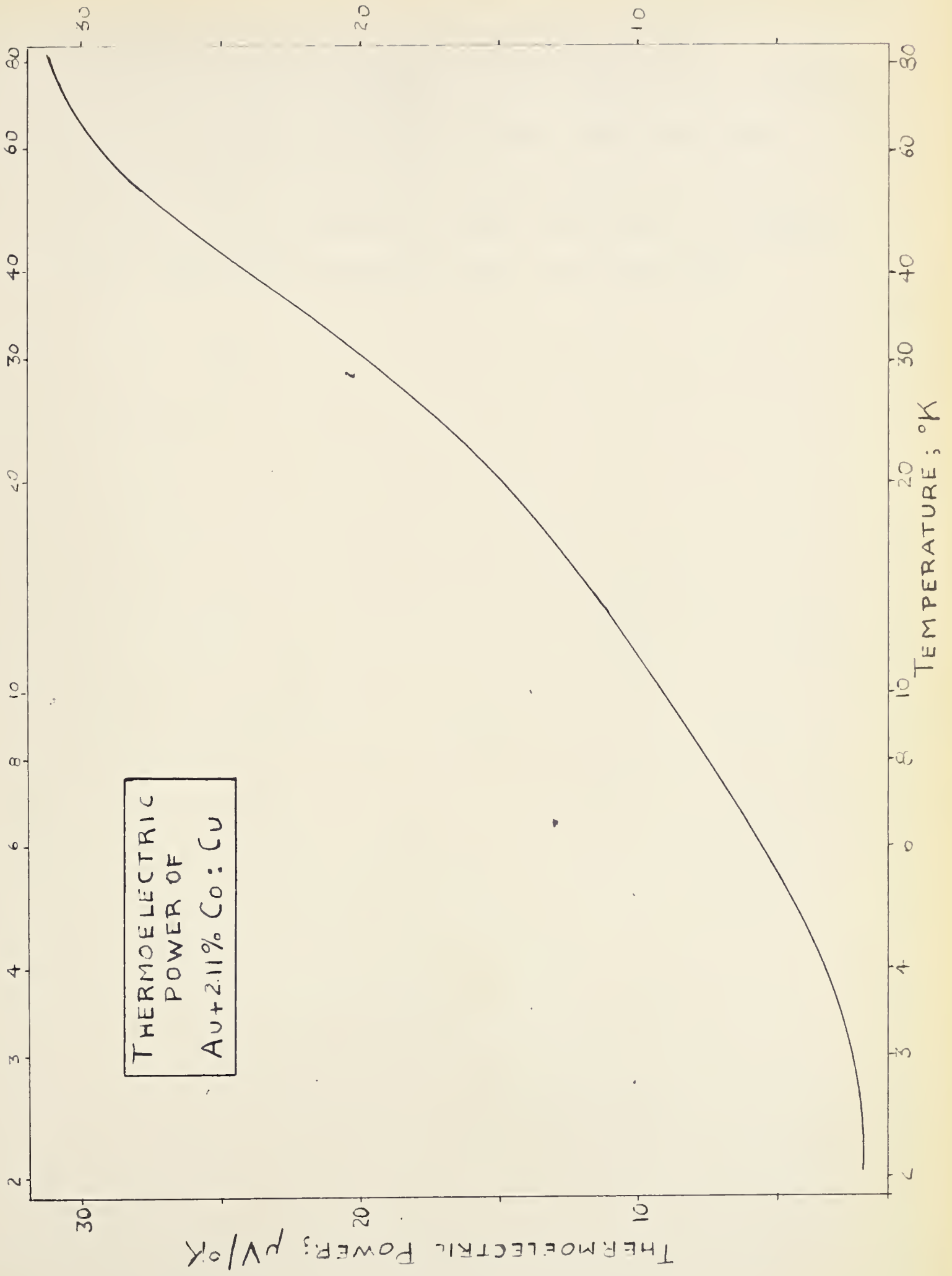


Figure 11.

are sensitive to purity and homogeneity and new calibrations may be necessary if new junctions are made using wires from a different source. The reference junction should be kept at a temperature as close as possible to the temperature being measured, to eliminate large temperature gradients along the alloy wires.

1.4 Measurement of Thermoelectric Power

In determining thermoelectric power one usually measures the thermoelectric force, then plotting this against temperature and differentiating the curve with respect to temperature one obtains the thermoelectric power of the two materials involved. In this instance, however, the thermoelectric power was obtained directly by applying a small gradient (less than 5% of the mean temperature of the specimen) and measuring the emf produced. In this way, the thermoelectric power relative to the specimen addenda (specimen holder and potential leads) was obtained. In order to obtain the absolute thermoelectric power of the sodium specimens the absolute thermoelectric power of the specimen addenda had to be found. To obtain this, a specimen of pure lead was mounted in the specimen holder and the thermoelectric power of lead against the specimen addenda was measured. The absolute thermoelectric power of the specimen addenda was then deduced using the values of Christian, Jan, Pearson and Templeton (1955) for the absolute thermoelectric power of lead. A full discussion of the transport properties of lead is given in Appendix I.

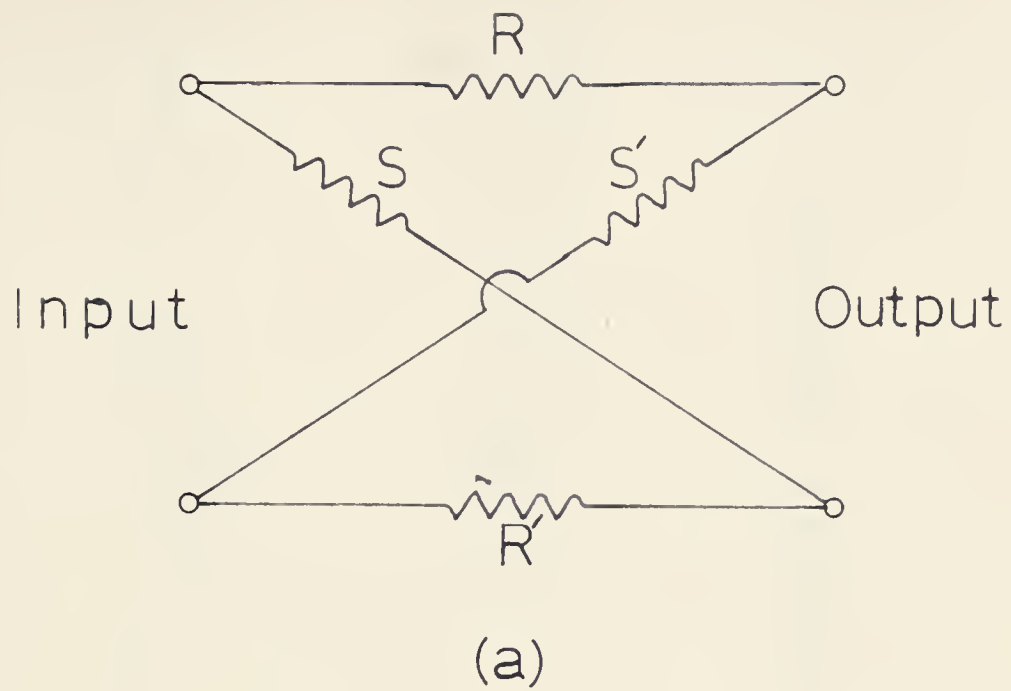
Rather large thermal voltages may be developed in the potential leads from the specimen if these leads extend from the very low temperature region up to room

temperature. In order to avoid errors introduced in this way a superconducting reversing switch was employed. This switch, which operates in a bath of liquid helium, enabled reversal of the emf from the specimen and its short potential leads; any thermal voltages in the subsequent wires which have gradients from 4°K to room temperature remain fairly constant during this reversal. By measuring the direct and reversed emf one is then able to obtain the actual voltage at the input of the superconducting reversing switch.

1.41 The Superconducting Reversing Switch

This reversing switch, shown in figure 12, was kindly provided by its originator, Dr. I. M. Templeton of the National Research Council. The operation of the switch is extremely simple in principle and has wide application in measuring very small voltages (of the order of 10^{-8} volts) at low temperatures provided that the source resistance is small.

The four resistances R and R', S and S', are small flat coils of tantalum, which become superconducting at 4.4°K. When the switch is sitting in a liquid helium bath (about 4.12°K) all four coils are superconducting. There are two small electromagnets each one of which can individually be energized by means of two coils (C). The first of these is used to provide a magnetic field



SUPERCONDUCTING REVERSING SWITCH

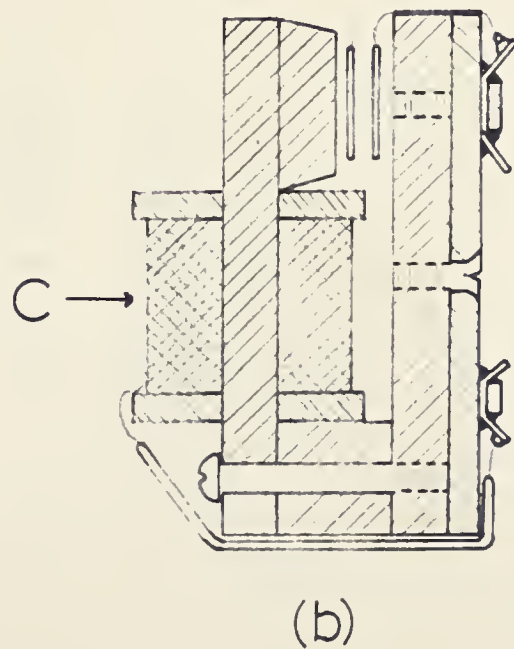
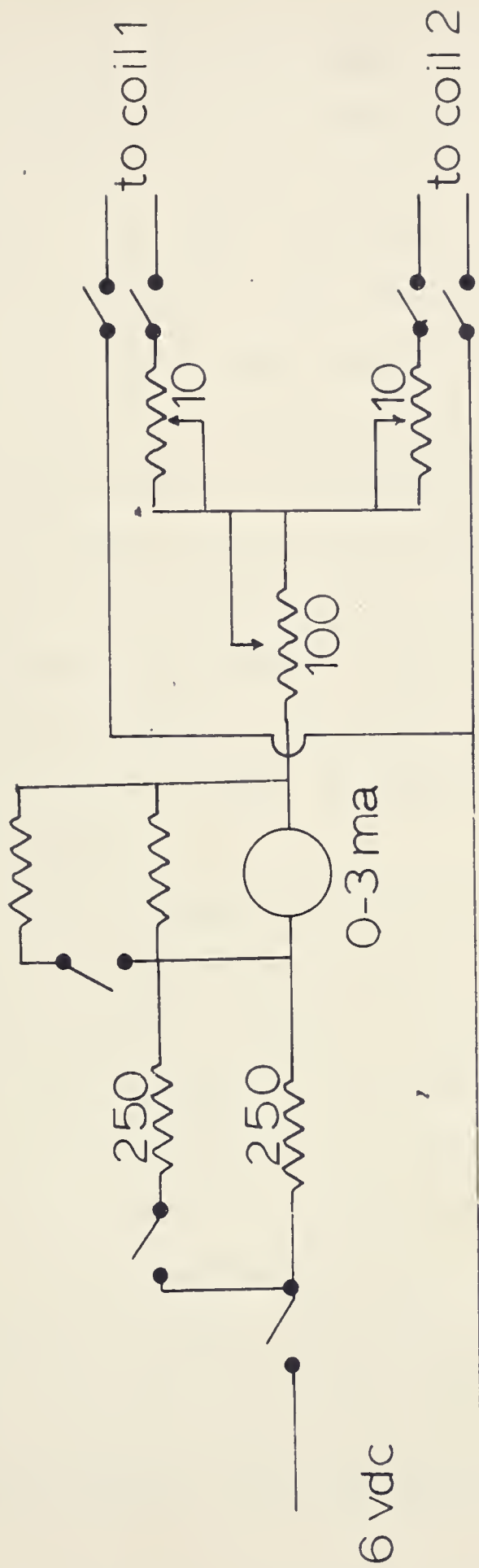


Figure 12



POWER SUPPLY FOR REVERSING SWITCH

Figure 13

which makes the coils R and R' return to the normal state (non-superconducting state) where they have a resistance of about 2 ohms each. Similarly coils S and S' can be changed from the superconducting to the normal state with the second magnet. A voltage applied across the input of the switch will appear across the output with the same magnitude but opposite polarity if R and R' are in the normal state while S and S' are superconducting. On the other hand, if S and S' are normal while R and R' are superconducting, the voltage across the input and output will be identical in both magnitude and polarity. The effect of the resistance of the two arms of the switch may be neglected since it is several orders of magnitude larger than the specimen resistance. #

The critical field required to change the tantalum coils to their normal state is about 60 gauss, which was produced with a current of about 30 ma in the coils of each electromagnet. The power supply used to energize the electromagnets is shown in figure 13. The switch itself was mounted on top of the outer can of the cryostat and the potential leads were brought to it from the specimen through a platinum-glass seal. This seal was made using 1/16" platinum tubing (rather than the usual

All specimens had resistances of less than 100 μ ohms.

platinum wire) so that the copper leads could pass through the seal without having to be cut and soldered at either side of the seal.

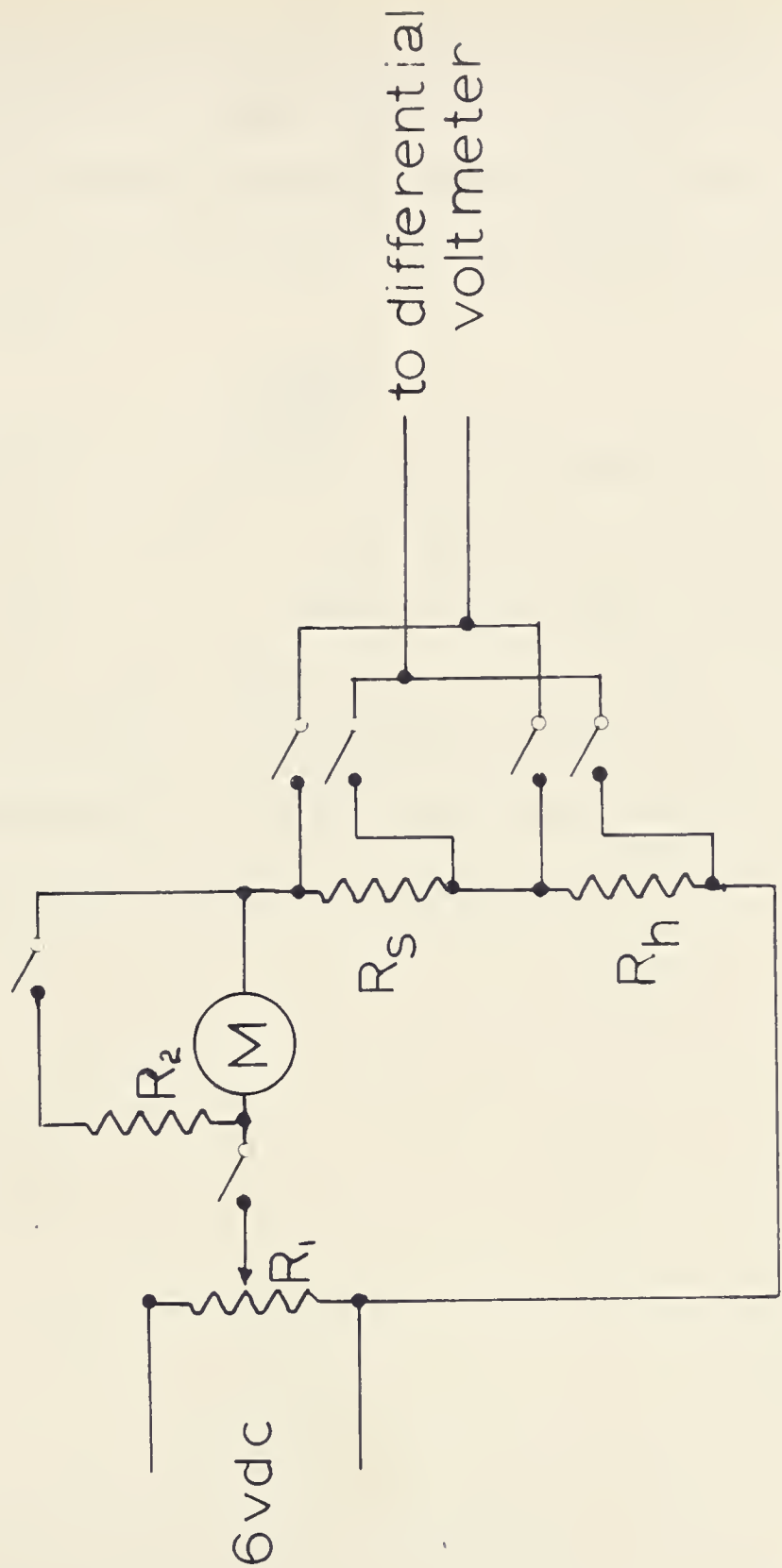
1.42 The Measuring Circuit

The voltage across the specimen was measured with the thermocouple measuring circuit described in section 1.33 since this circuit was already available. The resistance of the specimen could be measured by passing a known current through it and measuring the voltage difference between the ends of the specimen through the superconducting reversing switch.

1.43 The Specimen Heater

In order to produce a temperature gradient along the specimen a heater within the specimen chamber of the cryostat was required. This heater was made by winding about 450 ohms of #40 B&S gauge manganin wire on an insulated copper coil form to which it was cemented with glyptal baking varnish. The thermal link to the specimen was a short length of copper wire 0.040" in diameter, by which the coil form was suspended from the lower end of the specimen. The power supply for the heater is shown in figure 14. This power supply is constructed so that the power input to the heater can be measured.

This is necessary if one wants to measure the thermal



SPECIMEN HEATER SUPPLY

Figure 14.

conductivity of the specimen. R_h represents the heater in the diagram and R_s is a standard resistor of $300 \pm 0.15 \Omega$. If V_s is the voltage appearing across R_s and the voltage across the heater is V_h then the power input to the heater will be:

$$P = \frac{V_s V_h}{300} .$$

The differential dc voltmeter enabled measurement of voltages with an error of less than $10 \mu v$ to be made. Thus power input to the specimen was always determined with an error of less than 0.1%. It was found very convenient to measure the thermal conductivity along with the thermoelectric power since the only additional measurements required were V_s and V_h .

PART II

MEASUREMENTS OF THE THERMOELECTRIC POWER OF SODIUM

II REMARKS ON THE MARTENSITIC TRANSFORMATION AND THE THERMOELECTRIC POWER OF METALS

2.1 The Martensitic Transformation

Most crystallographic transformations take place by a mechanism of nucleation and subsequent growth of the nuclei by a diffusion process in which the growth of the new phase is achieved relatively slowly by a migration of the interphase boundaries. The atoms leave the old structure and attach themselves to the new structure aided in their diffusion by thermal agitation. Such a nucleation and growth transformation generates no noise.

This type of transformation cannot occur in sodium because the temperature at which the two phases (bcc and hcp) have the same Gibbs free energy is so low that diffusion cannot take place; hence the mechanism of the transformation must be a diffusionless one, in this case the martensitic mechanism.

In the martensitic process rearrangements of small groups of atoms, each keeping the same nearest neighbours, take place. Also coordinated movements of large numbers of atoms may occur within a time interval which is comparable to the time required for a sound wave to travel across the newly formed crystal. Hence the crystal may alter its structure abruptly and often with an audible click. When the temperature is low enough that the free

energy difference between the two phases favours the low temperature phase, regions of the high temperature phase will grow into regions with the low temperature structure by the process described above. As the temperature is lowered and the free energy difference increases, more regions are able to transform to the low temperature phase, until finally a low enough temperature is reached so that there is no longer an increase in the free energy difference between the two phases and the transformation stops. Usually the growth regions generate sufficient strain energy in the surrounding medium to stop complete transformation of the specimen. If the strain created during the growth of the nucleus is within the elastic limit when the growth ceases, the plate of the new phase will be in thermoelastic equilibrium. Such a plate can shrink or grow as the temperature is raised or lowered and in this way the transformation may be thought of as being reversible. More commonly however the strain produced during growth exceeds the elastic limit and plastic deformation results; the transformation then exhibits a temperature hysteresis.

The properties of the transformation are perhaps best explained if one considers figure 15. Upon cooling no crystallographic change takes place until temperature T_{α} is reached.

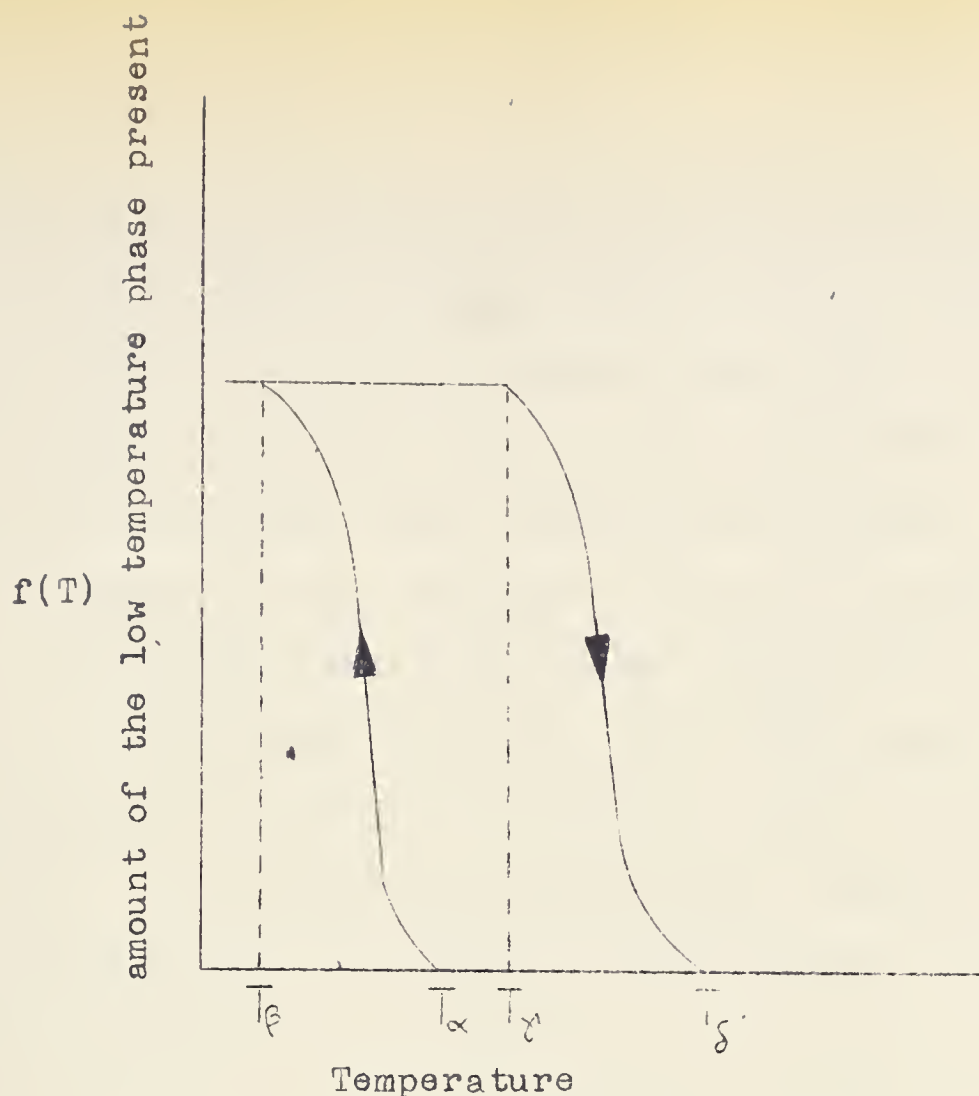


Figure 15. Schematic composition-temperature curve of a substance undergoing a martensitic transformation.

Upon subsequent cooling below T_α a fraction of the specimen transforms to the low temperature phase, the fraction depending on the temperature. Finally the transformation ceases when temperature T_β is reached and any subsequent cooling will have no effect on the fraction of the low temperature phase present. T_β may be interpreted as the temperature below which there is no further increase in the free energy difference between the two phases. If the specimen is now heated, no change in the fraction of the low temperature phase, $f(T)$,

occurs until a temperature T_δ ($T_\delta > T_\alpha$) is reached. Subsequent heating above T_δ causes $f(T)$ to diminish until finally $f(T)=0$ at a temperature T_s ($T_s > T_\delta$). There is another temperature, T_e ($T_s > T_e > T_\delta$), where the transformation may begin provided the specimen is suitably cold worked. It is to be noted that T_e is higher than T_α , the temperature at which the transformation would begin in the absence of cold work. T_e is probably the temperature at which the two phases have the same free energy. In sodium the above mentioned temperatures have been determined by Dugdale and Guban (1960) and Martin (1960) to be:

$$\begin{aligned} T_\alpha &\approx 36^\circ\text{K} \\ T_\delta &\approx 4^\circ\text{K} \\ T_s &\approx 45^\circ\text{K} \\ T_s &\approx 70^\circ\text{K} \\ T_e &\approx 51^\circ\text{K} \end{aligned}$$

It should be noted that these temperatures (particularly T_δ and T_s) do not agree completely with those given in the introduction which were obtained from Barrett (1956). The discrepancy is probably due to differences in samples and the fact that Barrett's data were obtained from X-ray measurements whereas the others are from electrical resistivity and specific heat measurements. In any case the only temperature of interest in the present work is T_α which seems to be about the same from all sources.

It can be seen from the foregoing discussion that the amount of transformation depends markedly on the

strain in the specimen. Purity, grain size and surface condition of the specimen also influence the amount of transformation but to a lesser extent. For example, an oxide layer on the surface might cause strain and thus reduce the amount of transformation.

2.2 The Electrical Resistivities of the Two Phases of Sodium

Dugdale and Guban (1960) have measured the effect of the martensitic transformation on the electrical resistivity of sodium. They found that:

- (1) the residual resistivity of sodium remained unchanged by the transformation.
- (2) Assuming that the measured resistivity was linearly dependent on f (the fraction of the hcp phase present), they were able to calculate the ideal resistivities of the two phases.

In calculating the ideal resistivities of the two phases they first assumed two extreme cases, namely that the two phases were in series and that they were in parallel. Fortunately the values obtained for each extreme did not differ greatly. They then proceeded to calculate the ideal resistivities of the two phases assuming that the resistivity measured for the mixture was that of a specimen composed of small spheres of hcp sodium embedded in a matrix of bcc sodium. This model provided them with values of ideal resistivities for the two phases which

differed by not more than a few percent from those obtained with either extreme model. In this way they have provided a method for determining the amount of transformation from measurements of the electrical resistance. We have used their resistivity data to determine the amount of transformation that took place in our specimens.

2.3 The Thermoelectric Power of Metals

The "free" electron theory of metals[#] has been used successfully to explain electrical resistivity and to a somewhat lesser degree thermal conductivity of metals. Sodium has thus far exhibited the best agreement between theory and experiment. Theory predicts that, provided the lattice vibrations are negligibly disturbed by either an electric field or temperature gradient and it is possible to define a mean free path for the electrons in a metal, the thermoelectric power will be given by (Mott and Jones 1936, p. 310):

$$S = \frac{\pi^2 k^2 T}{3e} \left(\frac{\partial \ln \sigma}{\partial \epsilon} \right)_{\epsilon = \epsilon_F} \quad (2.3-1)$$

where S is the absolute thermoelectric power, T is the absolute temperature, σ is the electrical conductivity as a function of the electron energy, ϵ , ϵ_F is the thermodynamic potential (Fermi energy) of the conduction electrons, k is Boltzmann's constant and e is the electronic charge. Unfortunately it is impossible to evaluate the above expression, even in the approximation in which the energy surfaces are spherical, without a detailed knowledge of the relaxation time (or mean free path) in the neighbourhood of the Fermi energy. That such knowledge is necessary can be seen from the

[#] The electrons are regarded as quasi-free, their energy being $\epsilon(\vec{k}) = \hbar^2 |\vec{k}|^2 / 2m^*$ where \vec{k} is the electron wave vector and m^* is the effective mass of the electron.

expression below:

$$\frac{\partial \ln \sigma}{\partial \epsilon} = \frac{\partial \ln \Lambda}{\partial \epsilon} + \frac{\partial \ln A}{\partial \epsilon}, \quad (2.3-2)$$

where Λ is the electron mean free path and A is the area of the Fermi surface. Two limiting cases have been worked out.

- (a) In the temperature range where the scattering of the electrons is due primarily to lattice vibrations S should be given by:

$$S = \frac{\pi^2 k^2 T}{e \epsilon_F}. \quad (2.3-3)$$

- (b) In the temperature range where the scattering of the electrons is due primarily to impurities (impurities are treated as point imperfections) S should be given by:

$$S = \frac{\pi^2 k^2 T}{3e \epsilon_F}. \quad (2.3-4)$$

It can be seen from (2.3-3 and 4) that S varies linearly with T and this corresponds with observed results at least for sodium. It has been found experimentally by MacDonald and Pearson (1953, 1954) and by MacDonald, Pearson and Templeton (1958) that at low temperatures, where impurity scattering is dominant, the thermoelectric power of sodium is about an order of magnitude larger than predicted by (2.3-4).

It is evident from equations (2.3-3) and (2.3-4) that the sign of the thermoelectric power depends on the sign

of the charge carriers. Thus for metals in group I of the periodic table one would expect negative thermoelectric powers since there is no evidence of hole conduction. However positive thermoelectric powers have been observed in the noble metals and some of the alkali metals.

Thus the "free" electron theory of thermoelectric power fails almost completely to account for the observed thermoelectric power in metals. It seems that the reason for this failure is not due so much to lack of understanding of the mechanisms involved, as it is to the nature of the assumptions made in order to obtain mathematical simplicity. A qualitative description of the mechanisms giving rise to thermoelectric effects will be given below.

Consider a single conductor under the influence of a temperature gradient. The electrons would tend to drift from the hot to the cold end in a manner analogous to an ordinary molecular gas. This drift will establish a potential difference in the conductor, the cold end becoming more negative by virtue of an excess of electrons. This excess electron density at the cold end exerts an electrostatic repulsion which serves to bring the process to a halt. This type of mechanism gives rise to one component of thermoelectric power which may be referred to as the "diffusion thermoelectric power". In deriving equation(2.3-1) it is assumed, in order to simplify mathematically the solution of the Boltzmann transport

equation, that the distribution function of the lattice may be replaced by its equilibrium value.

Gurevich (1945, 1946) pointed out that the assumption that the phonons, quanta of lattice vibrational energy, are in thermal equilibrium may in fact account for some of the failure of the "free" electron theory. He suggested that under the influence of a temperature gradient the phonons might also drift from the hot to the cold end giving rise to a "phonon current". Some of these phonons would suffer collisions with conduction electrons and transfer momentum to these electrons thus "dragging" the electrons with them. This mechanism gives rise to another component of thermoelectric power, "the phonon drag". The magnitude of the phonon drag component will depend intimately on the strength of the phonon-electron interaction in the metal.

Recently, Hanna and Sondheimer (1957) have considered this problem and produced a theory which provides much more reasonable results, at least for sodium where they differ by only about 10% from the experimental values in the temperature range around 40°K. It should be noted however that both the diffusion thermoelectric power and the phonon drag thermoelectric power arise from electrons moving down the temperature gradient thus giving rise to negative thermoelectric powers only.

M. Bailyn (1958) suggested that the sign of the

phonon drag thermoelectric power is not invariable in principle if one considers the so called electron-phonon "Umklapp" processes which were proposed by Peierls (1930) and Bardeen (1937). In such "Umklapp" processes an electron absorbs a phonon thereby arriving in some state at the boundary of the Brillouin zone whereupon it suffers a Bragg reflection. Thus it is possible for electrons having undergone an "Umklapp" process to be in a state with wave vector such that they are moving up the temperature gradient and give rise to a component of thermoelectric power which will be positive in sign.

J. M. Ziman (1959) published a theory in which he considered phonon drag (both the normal and "Umklapp" processes) and was able to obtain good results for sodium assuming that the Fermi surface is spherical. His theory takes into account the anisotropy of the Fermi surface, in the case of the other alkali metals, and indicates that the thermoelectric power is sensitive to the anisotropy of the Fermi surface. One might expect this since the relative frequency of "Umklapp" processes must be intimately related to the proximity of the Fermi surface to the zone boundary.

III EXPERIMENTAL METHOD

3.1 Preparation of Sodium Specimens

The alkalis require somewhat more care in handling than do other metals as they are more highly reactive. Before preparing any of the specimens some benzene and liquid paraffin (trade name "Stanolax") were dried by adding small pieces of sodium to each until no further oxidation occurred. All preparation took place while the specimens remained immersed under these dried solutions in order to avoid exposure to the air as sodium oxidizes very rapidly. Eight sodium specimens were prepared but only the last three of these: Na-6, Na-7 and Na-8 were used in the experiments as difficulty was encountered in mounting the other five specimens.

Na-6 was obtained from A. D. Mackay Inc., New York packaged in evacuated glass vials each containing about two grams of sodium. A vial was broken under dry "Stanolax" and small pieces of sodium were pried out of the vial and placed in the cylinder of the extruder described in appendix II. The sodium was subsequently extruded as a wire about 1 mm in diameter, the extrusion being done directly into another dry "Stanolax" bath.

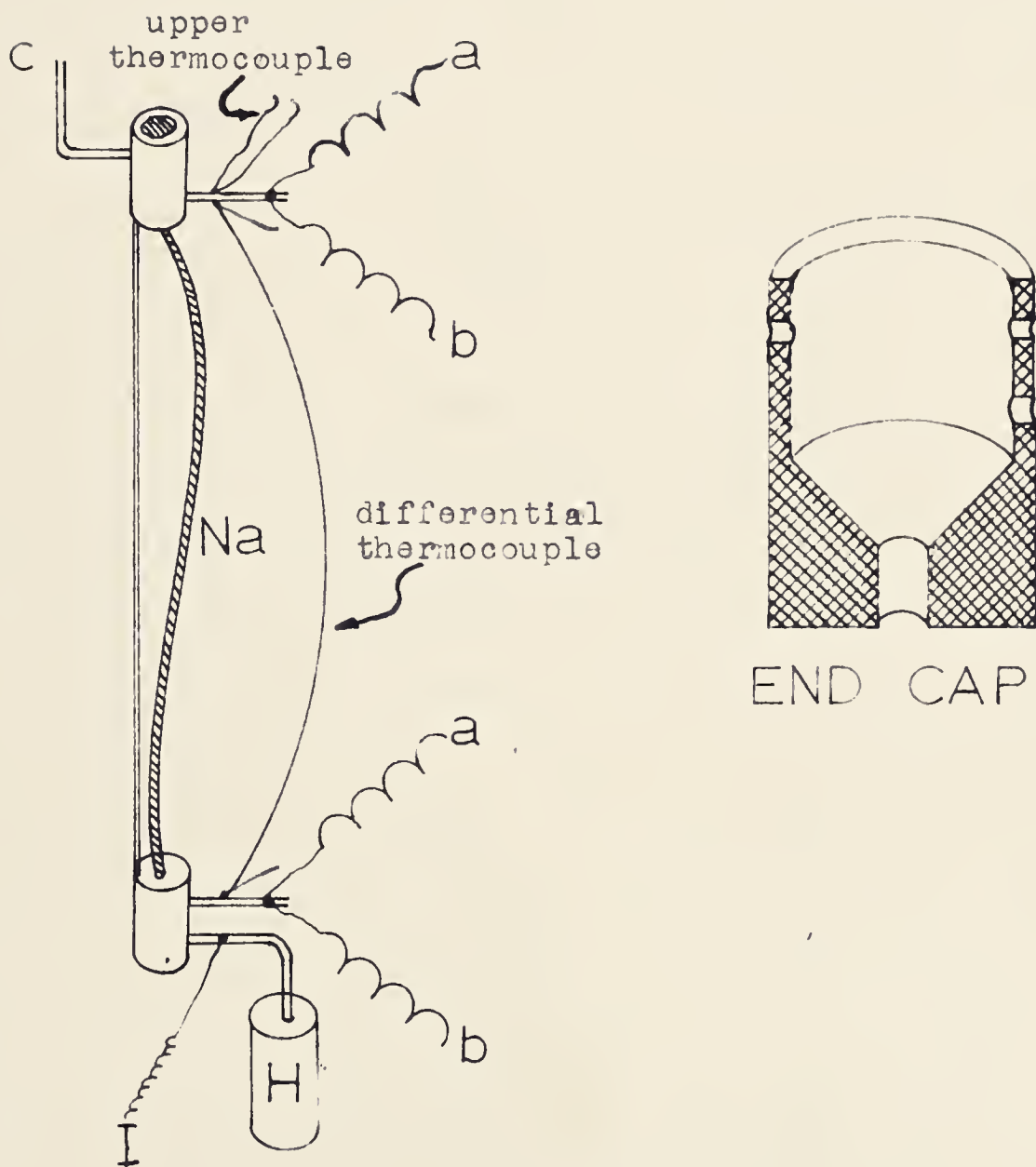
Na-7 and Na-8 were obtained from the Physics Department stock; the original source is unknown. The sodium was melted in an inert atmosphere using the method described

by MacDonald (1956). The sodium was then cast into a vial and sealed. From this point on the same procedure was followed as is described for Na-6.

3.11 Mounting of the Sodium Specimens

Until recently measurements of transport properties of the alkali metals have been performed on specimens which were cast in glass capillaries. However, because of the nature of the martensitic transformation, which may be inhibited by strain in the specimen, it was necessary for this investigation to mount the sodium as a free specimen. Since sodium is very soft (its consistency is similar to plasticine) a special specimen holder had to be constructed. The specimen holder and method of mounting are shown in figure 16. The specimen holder consists of two copper end caps joined by a piece of stainless steel. The extruded sodium wire was placed in the holder with about a $\frac{1}{8}$ " length of sodium passing through each end cap. The end caps were subsequently filled with sodium to which enough pressure was applied to cold weld it to the other pieces of sodium in the cap.

The specimen holder filled with sodium was attached to the top of the specimen chamber by a 0.040" diameter piece of copper wire, c, which was soldered to the specimen chamber at the top end while the other end of the wire was pushed into the sodium through a hole in



SPECIMEN MOUNTING

Figure 16.

the specimen holder cap. The specimen heater, H, was attached to the bottom of the specimen in a similar way. Current could be supplied to the specimen through a copper wire, I, which was attached to the copper wire supporting the heater. The electrical contact between the specimen and the top of the specimen chamber served as the current return circuit.

One junction of each of the thermocouples (upper and differential) was attached to a 1" length of 0.032" diameter copper wire with fingernail varnish. A piece of cigarette paper between the thermocouples and the wire provided electrical insulation while maintaining good thermal contact. A copper lead, a, from the superconducting reversing switch and a potential lead, b, of copper with a manganin section in it were soldered to the 1" piece of wire to which the thermocouple junctions had already been attached. This wire was pushed into the upper specimen holder cap as shown in figure 16. The manganin section in the potential lead served as a thermal dam since the thermal conductivity of such an alloy is appreciably lower than that of a pure metal such as copper. The other junction of the differential thermocouple and the second lead from the superconducting reversing switch, as well as the second potential lead were attached to the bottom of the specimen holder in the same way as described above. The other junction of the upper

thermocouple was anchored to a copper post which provided good thermal contact with the reference bath in the dewar.

In order to minimize oxidation of the specimen, the sodium was mounted in the specimen holder under oil. Then the specimen holder and sodium were mounted in the cryostat and the oil dissolved with sodium-dried benzene just before the cans were put on the cryostat. The time required between the application of the benzene and the evacuation of the cryostat was only about two minutes. The specimen chamber was allowed to out gas for about one hour at room temperature and then the cryostat and specimen were cooled to liquid air temperature. All specimens were mounted on the evening preceding the experiment and maintained at liquid air temperature overnight.

3.2 Experimental Procedure

After the specimen had been cooled to liquid air temperature, the inner chamber was filled with liquid air through the needle valve. The liquid air dewar was then removed and was replaced with an empty liquid helium dewar. Another larger dewar filled with liquid air was placed around this dewar. The next step was to cool the cryostat to about 60°K by pumping the liquid from the inner chamber. When the liquid had all evaporated the exchange gas was pumped from the specimen chamber and outer can. Liquid helium was then syphoned into the inner dewar cooling the outer can to 4°K . At this point the procedure became somewhat different from the conventional method of allowing exchange gas to enter the cryostat which would cool the specimen to 4°K . Because measurements on pure bcc sodium were required the specimen was not allowed to cool below 36°K . To produce the required slow cooling which could be stopped with ease at some temperature above 36°K (usually in these experiments we performed the first measurement at about 43°K), the temperature controller was pre-set to about 40°K and the needle valve was opened slightly while the inner chamber was being pumped. The cold helium gas passing through the inner chamber cooled it until the desired temperature was reached. At this time the needle valve was again closed and the controller

maintained the cryostat at this temperature. The specimen resistance was determined at this temperature. Then the lower end of the specimen was heated and when thermal equilibrium was reached the thermoelectric power of the specimen with respect to the specimen addenda (specimen holder and potential leads) was measured by the methods described in part I. Similar measurements were carried out at lower temperatures until the specimen was cooled to 4°K . The specimen was then heated to various temperatures using the electronic temperature controller until the initial temperature (about 43°K) was reached. Temperatures at which measurements were taken on the heating part of the cycle were chosen to correspond as nearly as possible to those used on the cooling part of the cycle to facilitate comparison of the results. The above description applies to measurements on Na-6 and Na-7.

It was suggested by Dr. J. S. Dugdale that it would be worthwhile to ascertain that the change in thermoelectric power was indeed due to the structural change produced by the martensitic transformation and not due to the presence of additional scattering centers such as dislocations produced by the transformation. An experiment was therefore carried out on Na-8 in which the specimen was first cooled to 43°K and the thermoelectric power of the pure bcc phase was measured. It was then cooled to 4°K to allow part of the specimen to transform and subsequently

was heated to 83°K where the whole specimen would again become pure bcc'. However any dislocations introduced by the transformation could not anneal out at such a low temperature. Finally the specimen was allowed to cool to 43°K where its thermoelectric power was again measured.

3.21 Comments on the Measurements and Calculations

(a) Ideal Electrical Resistivity

The resistance of the specimen was obtained by measuring the potential appearing between the ends of the specimen due to a known current flowing through the specimen. The average potential obtained from measurements with the current flowing in each direction was used in order to eliminate the effect of stray thermoelectric voltages present in the specimen and potential leads. The ideal resistance of the specimen was obtained using Matthiessen's rule, $R = R_1(T) + R_0$, where R is the measured resistance, $R_1(T)$ is the component of resistance due to scattering of conduction electrons by the thermal vibrations of the lattice and R_0 is the component of resistance due to scattering of conduction electrons by impurities. Experimentally R_0 was determined at 4°K because at this or lower temperatures $R_1(T) \ll R_0$. The resistance of the specimen holder was so large that its effect on the measurements was negligible for reasons explained in appendix I. To convert resistance to resistivity the dimensions of the specimen had to be known. The

geometrical factor l/A , where l is the length of the specimen and A is the cross-sectional area, is difficult to measure directly. Instead it was deduced from a measurement of the ideal resistance at about 40°K (obtained during the first cooling cycle of the sample) and the values for the ideal resistivity of pure bcc sodium obtained by Dugdale and Guban (1960), using the relation:

$$\frac{l}{A} = \frac{R_i(T)}{\rho_i(T)}.$$

(b) Thermoelectric Power

The arithmetic mean of the temperatures of the end points of the specimen (points where the potential leads were inserted) was taken as the temperature at which the thermoelectric power of the specimen was measured. The temperature difference between the ends of the specimen was usually 2 to 3 percent of the mean temperature of the specimen. The absolute thermoelectric power of the specimen was derived from the measured thermoelectric power of the specimen and addenda using values obtained for the thermoelectric power of the specimen addenda as is described in appendix I.

(c) Thermal Conductivity

The thermal conductivity of the specimens was deduced by measuring the power input to the specimen heater in the way described in section 1.43. The shape factor, l/A , obtained from the electrical resistance data, was used to calculate the thermal conductivity.

IV RESULTS AND DISCUSSION

4.1 The Absolute Thermoelectric Power of Hexagonal close-packed sodium at 43°K

In order to deduce the absolute thermoelectric power of the hexagonal close-packed phase two simple models were used. In the first of these it was assumed that the thermoelectric force of the two phases would add in a way similar to two batteries in series, while in the second model it is assumed that one can treat the two phases as batteries in parallel. The absolute thermoelectric power of the hexagonal close-packed phase can be expected to lie somewhere between the values deduced from these two extreme models. The value obtained from the series model however, is expected to be a better approximation to the actual thermoelectric power of the hcp phase for reasons which will be outlined in section 4.13. The results obtained are given in Table I.

4.11 The Series Model

Consider a specimen in which the distribution of the two phases is as shown in figure 17 (a). If one then measures the resistivity between A and C at a particular temperature one can then calculate the fraction, f , of the hcp phase present using the values obtained by Dugdale and Guggan (1960) for the ideal resistivity of the hcp phase, ρ_{hcp} , and the ideal resistivity of the bcc phase,

ρ_{bcc} . If ρ^* is the ideal resistivity of a mixture of the two phases then the fraction f of the low temperature phase will be given by:

$$f = \frac{\rho_{bcc} - \rho^*}{\rho_{bcc} - \rho_{hcp}}$$

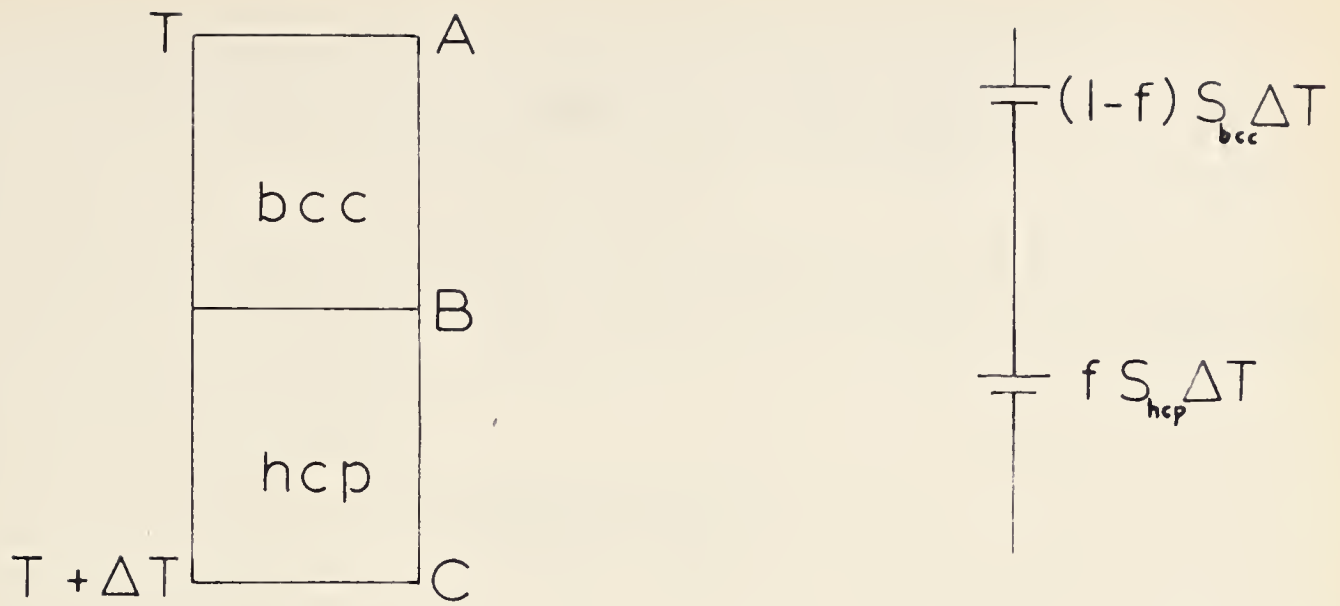
Consider a temperature difference, ΔT , between A and C. If the thermal conductivities of the two phases are the same (results of measurements on these samples indicate that this is indeed a good approximation) then there will be a temperature difference $(1-f)\Delta T$ between A and B and a temperature difference $f\Delta T$ between B and C, thus the thermoelectric power of the hcp phase will be given by:

$$S_{hcp} = \frac{S^* - (1-f)S_{bcc}}{f}$$

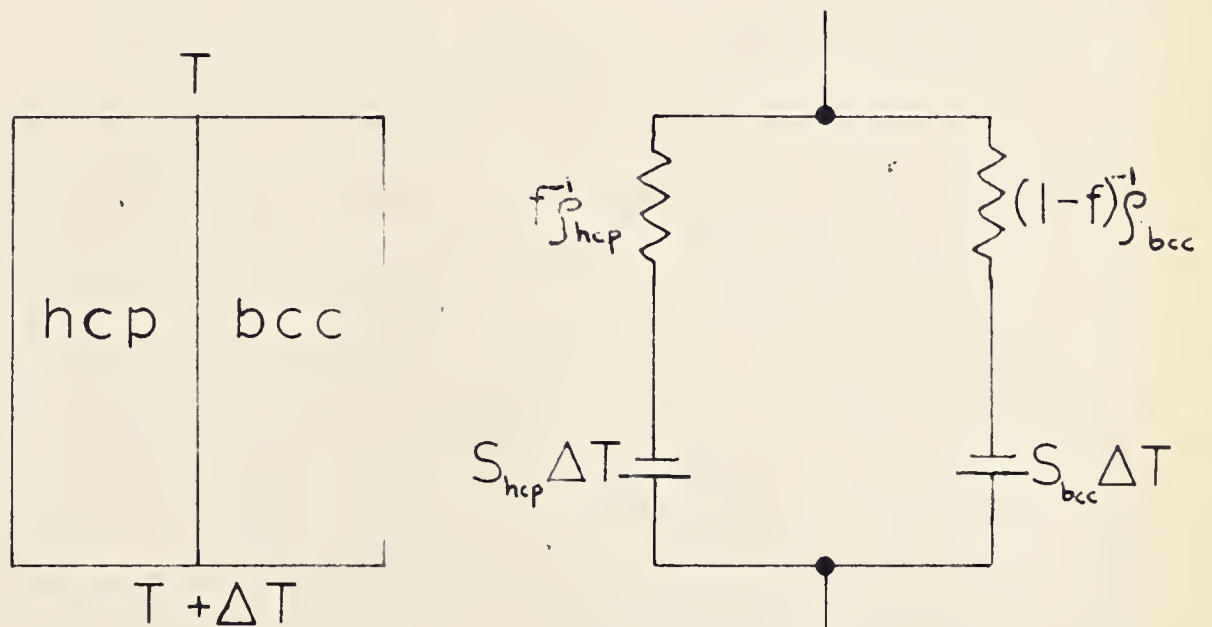
where S^* is the measured thermoelectric power of the mixture of the two phases and both S^* and S_{bcc} are measured at the same temperature.

4.12 The Parallel Model

Consider the parallel model in figure 17 (b). If it is assumed that the thermoelectric force due to each phase may be represented by a battery having an internal resistance equal to the resistance of the particular phase then the circuit shown below forms an adequate model and the thermoelectric power of the hcp phase



(a) Series Model



(b) Parallel Model

Figure 17.

will be given by:

$$S_{hcp} = S_{bcc} + (S^* - S_{bcc}) \frac{(1-f)^{-1} S_{bcc} + f S_{hcp}}{(1-f)^{-1} S_{bcc}}.$$

If this model is valid, the fraction, f , must be calculated assuming that the resistivities add in parallel as well and hence:

$$f = \frac{S_{hcp} (S_{bcc} - S^*)}{S^* (S_{bcc} - S_{hcp})}.$$

4.13 Analysis of the Results

Experimental values obtained for the absolute thermoelectric power and the ideal electrical resistivity of Na-7 are shown on the graphs of figures 18 and 19. The results obtained for the absolute thermoelectric power of the hcp phase are given in the form of ratios $\frac{S_{hcp}}{S_{bcc}}$ in table I. It has been noted by many observers, such as MacDonald et al. (1953, 1954, and 1960), Pearson and Templeton (1955), that the thermoelectric power of metals often varies by a few percent from one specimen to another. This variation is probably due to impurities present. It is for this reason that the thermoelectric power of the hcp phase is given as a ratio. Dugdale and Guban (1960), by comparing results of other workers, have pointed out that for large amounts of transformation such as we have obtained, the average grain size would probably be limited by the cross-sectional area of the

Table I SUMMARY OF RESULTS

Specimen	Na 6	Na 7	Na 8
Shape Factor l/A (cm^{-1})	603	894	
Residual Resistivity [#] Ratio	3×10^{-3}	1.5×10^{-3}	
Fraction of hcp phase present (series model)	0.26	0.41	0.75 ^{##}
Fraction of hcp phase present (parallel model)	0.25	0.37	
Thermoelectric Power of the bcc phase at 43°K ($\mu\text{V}/\text{deg}$)	-1.87	-1.90	-1.82 -1.83 ^{###}
Thermoelectric Power of the Mixture at 43°K ($\mu\text{V}/\text{deg}$)	-1.83	-1.82	-1.67
Ratio of Thermoelectric Power of the two phases $\frac{S_{\text{hcp}}}{S_{\text{bcc}}}$ at 43°K (series model)	0.90	0.91	
Ratio of Thermoelectric $\frac{S_{\text{hcp}}}{S_{\text{bcc}}}$ Power of the two phases $\frac{S_{\text{hcp}}}{S_{\text{bcc}}}$ at 43°K (parallel model)	0.92	0.90	

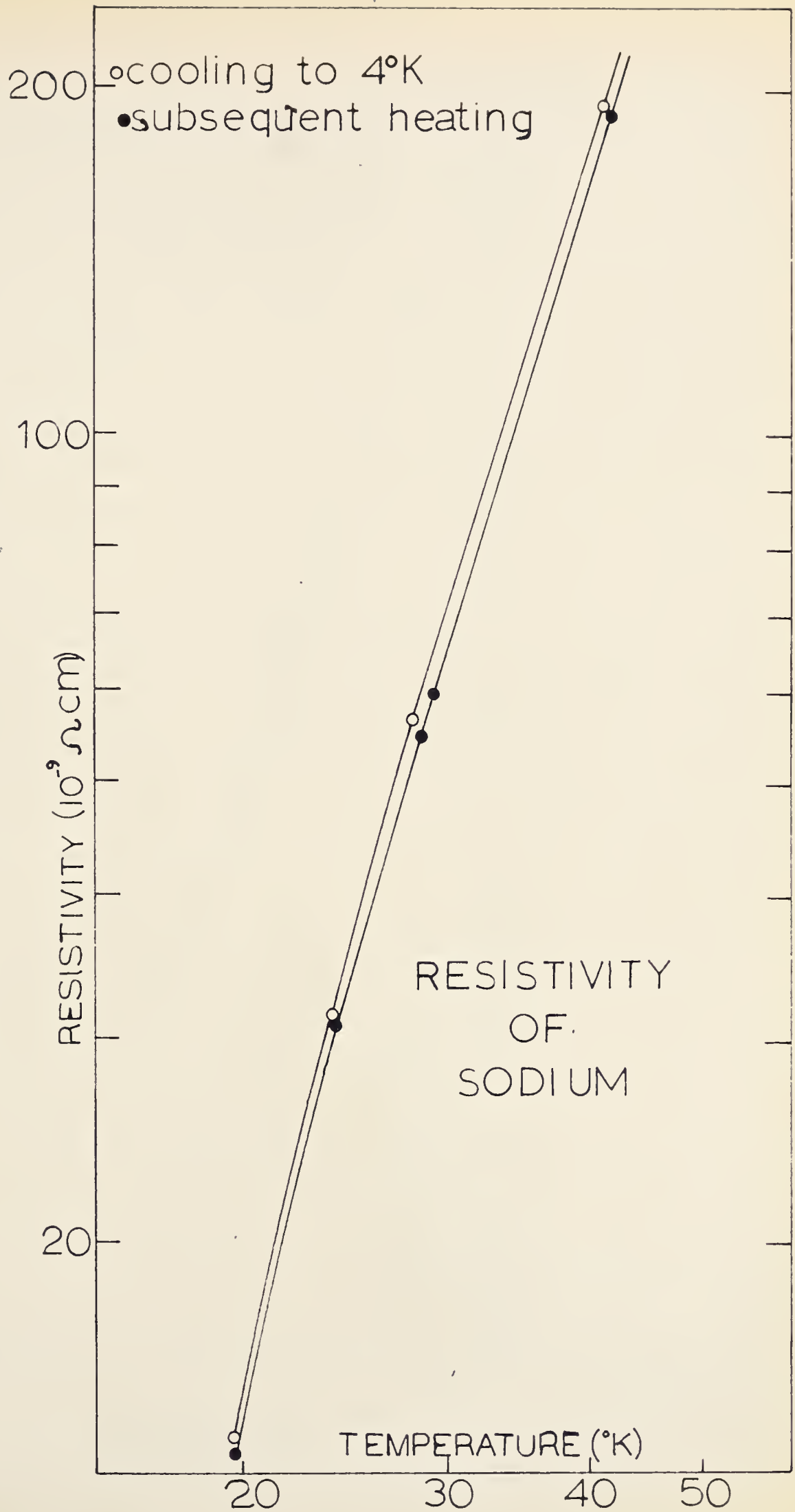
Assuming Hackspill's (1910) value for the resistivity of sodium at the ice point.

Estimated from thermoelectric power measurements using the series model.

This value was obtained after heating the partially transformed specimen to 83°K (6°K above the reversion temperature) and then allowing it to cool to 43°K at which temperature its thermoelectric power was measured.

specimens. Regions of each phase would then extend completely across the specimen. It is therefore rather gratifying that the values deduced from the series model should agree so well with one another.

Unfortunately it was not possible to measure the electrical resistivity of Na-8 as the lead which supplies current to the specimen burned out while we were trying to take a residual resistance measurement. The amount of transformation was however calculated from the thermoelectric power measurements using the series model and was found to be about 75%. The large amount of transformation is not surprising. It was noticed that when this specimen was extruded the resulting sodium wire had kinks in it. The angle between two adjacent straight segments was about equal at each kink. This strongly suggests that recrystallization was occurring at room temperature. The kinks are probably at crystal boundaries which tend to occur at certain specific angles which minimize the lattice misfit across the boundaries. Such recrystallization occurs only in extremely pure sodium. It is interesting to note residual resistivity measurements on another specimen cast into a glass capillary from the same melt gave $\rho_0/\rho_{273} = 5 \times 10^{-4}$, which indicates very high purity sodium.



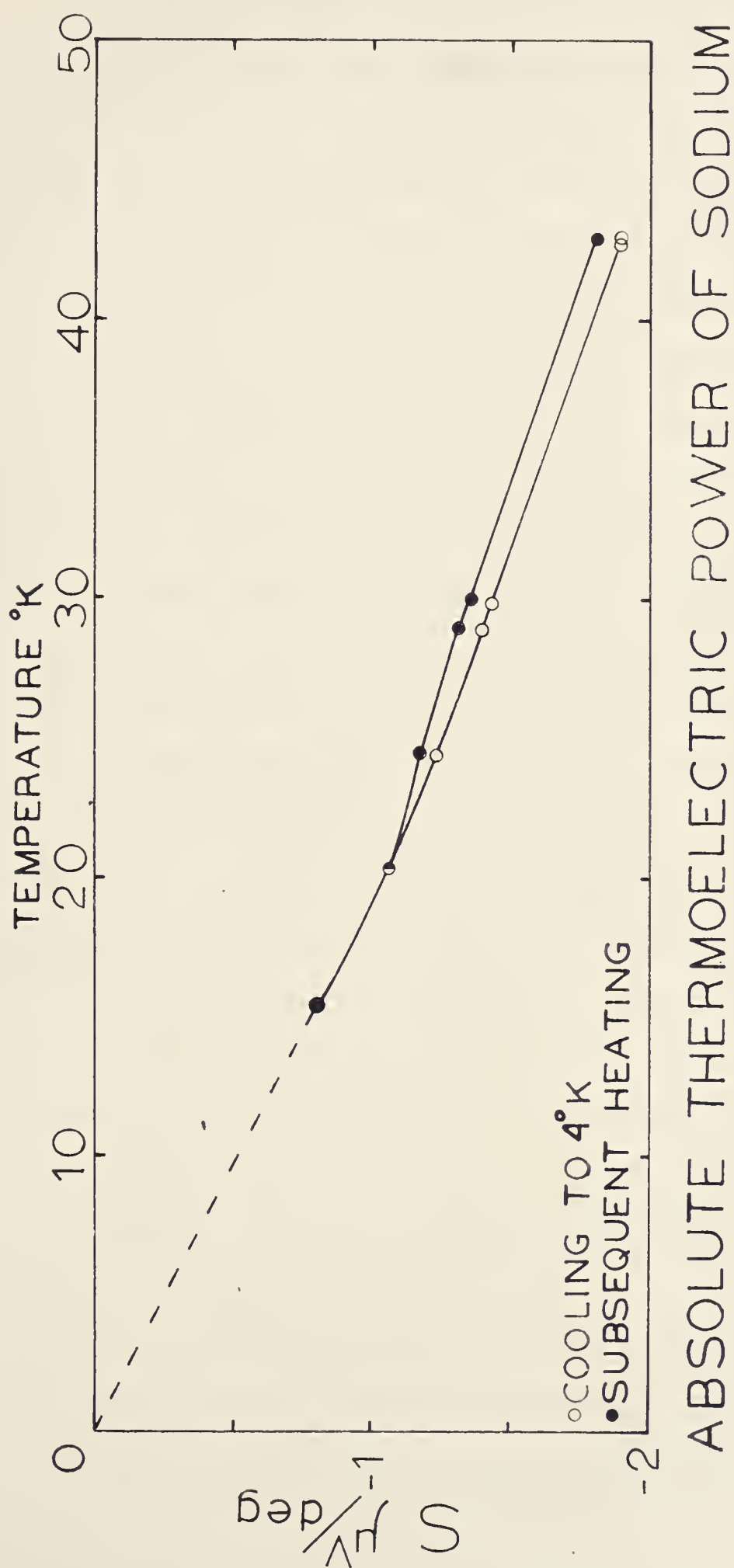


Figure 19.

4.2 Conclusions

The change in the thermoelectric power of sodium is indeed due to the structural change produced by the martensitic transformation rather than to the introduction of additional scattering centers such as dislocations. This is evident from the fact that there was no appreciable change in the thermoelectric power of the pure bcc phase of sodium after one transformation cycle. The thermoelectric power of the hcp phase of sodium is lower in magnitude than that of the bcc phase by about 10%.

Measurements by Dugdale and Guban (1960) have shown that the residual resistivity of sodium remains unaltered by the transformation and they have interpreted this as indicating that the Fermi surface remains unchanged in going from bcc to hcp structure. Hence there is no change in Fermi energy and the diffusion component of thermoelectric power would also remain unchanged. Thus the lower thermoelectric power of the hcp phase must arise from a decrease in magnitude of the net phonon drag component of thermoelectric power. This indicates that the ratio of "Umklapp" to normal processes in electron-phonon interactions must be larger in the hcp phase than in the bcc phase.

Dugdale and Guban (1960) have also discussed the Brillouin zone structure of the hcp phase and pointed out that in the case of the two hexagonal faces the

energy gap for the conduction electrons is either zero or very small. On the other hand in the case of the six rectangular faces the distance from the Fermi sphere to the zone boundary is about 4% of the distance from the zone face to the center of the zone. In contrast, the comparable figure in the bcc structure is about 12%. This suggests that Umklapp processes would indeed be more frequent in the direction of the six rectangular faces.

Therefore the change of the thermoelectric power of sodium by the martensitic transformation may be interpreted as being due to the closer proximity of the Fermi surface to the zone boundary in the hcp phase as compared with the bcc phase.

Since present theory predicts an appreciable phonon drag contribution to the thermoelectric power of sodium it would be of interest to investigate the electron-phonon interaction experimentally by more direct methods than have been attempted to date. One might for example excite the lattice vibrations in a metal by ultrasonic techniques thus giving rise to a phonon current and attempt to determine if this gives rise to a potential difference between the ends of the specimen. Such a potential difference would give direct evidence of the phonon drag, as well as providing valuable information

about the phonon-electron interaction particularly if this experiment were carried out at various frequencies. If such a potential difference could be measured as a function of phonon wavelength it in fact would provide some estimate of the relative strengths of normal and "Umklapp" processes in phonon-electron collisions thus giving a firmer foundation to the present theories.

APPENDIX I

THE ABSOLUTE SCALE OF THERMOELECTRIC POWER AND THE TRANSPORT PROPERTIES OF LEAD

Thermoelectric Effects

When two dissimilar conductors are joined together at both ends and the two junctions are kept at different temperatures, an emf is set up. This emf which is roughly proportional to the temperature difference is known as the Seebeck emf. The Seebeck effect may be considered as being due to two other effects: the Peltier effect and the Thomson effect.

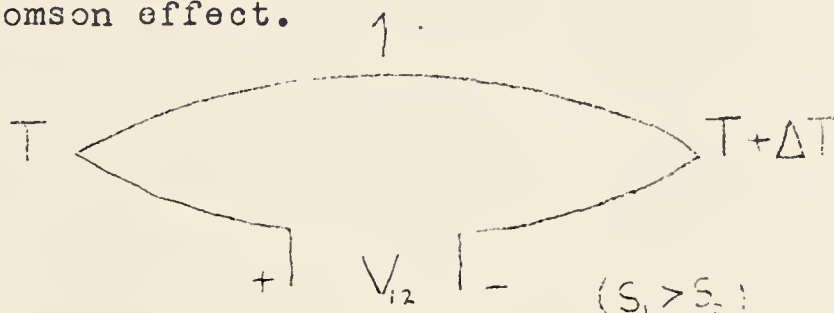


Figure 20. Thermoelectric Circuit

The first of these will occur when two dissimilar metals are joined together and kept at a constant temperature while a current flows through the junction. In addition to the Joule heat generated by the current, heat is generated or absorbed reversibly at the junction. The heat developed at the junction is proportional to the current so we can define the Peltier coefficient Π_{12}

as the heat emitted per second due to a unit current flowing from conductor 1 to conductor 2 (figure 20).

As the Peltier effect is reversible $\Pi_{12} = -\Pi_{21}$.

The Thomson effect is also a reversible effect but it occurs in a single conductor. When a current I passes between two points of a homogeneous conductor which have a temperature difference ΔT between them, an amount of heat $\mu I \Delta T$ is emitted or absorbed in addition to the Joule heat. μ is the Thomson coefficient of the metal which forms the conductor and may be thought of as being essentially a specific heat per unit charge.

The Kelvin Relations

The Kelvin relations which give the thermoelectric power in terms of the Thomson and Peltier effects can be stated as follows:

$$T \frac{dV_{12}}{dT} = \Pi_{21}$$

and

$$T \frac{d^2 V_{12}}{dT^2} = \mu_1 - \mu_2.$$

The sum of the thermoelectric voltages around the circuit (V_{12} in figure 20) is called the thermoelectric force or Seebeck voltage. One usually defines another quantity the thermoelectric power, S ,

$$S_{12} \equiv \frac{dV_{12}}{dT}$$

and then the Kelvin relations may be written

$$T S_{12} = \Pi_{21}$$

$$T \frac{dS_{12}}{dT} = \mu_1 - \mu_2.$$

S_{12} may be thought of as the entropy difference per unit charge between the two conductors and one can thus write:

$$S_{12} = S_1 - S_2,$$

where S_1 and S_2 are now the "absolute thermoelectric powers" of the two conductors. Thus one can write the absolute thermoelectric power as:

$$S = \int_0^T \frac{\mu}{T} dT.$$

At first sight this may seem a difficult parameter to determine as it involves an extrapolation to $T = 0^\circ\text{K}$. It should be noted however that μ is zero for a superconductor. Thus, in principle, the absolute thermoelectric power of any metal can be determined at low enough temperature with a superconductor as the other element of the couple, but at higher temperatures it is necessary to measure the Thomson heat of the metal.

The Thermoelectric Power of Lead

Measurements of the absolute thermoelectric power of a metal are usually made by measuring the thermoelectric power of the metal against lead either directly or indirectly. A knowledge of the absolute thermoelectric power of lead is then necessary. Until recently the absolute scale of thermoelectric power of lead used was that determined by Borelius, Keesom, Johansson and Linde (1932). This scale is however somewhat in error at low temperatures. A careful redetermination was made by Christian, Jan, Pearson and Templeton (1958). They

measured the absolute thermoelectric power of lead against Nb_3Sn , an alloy which remains a superconductor up to 18°K , then using measured values for the Thomson heat of lead above 20°K obtained the present scale of thermoelectric power.

Lead is particularly well suited as a thermoelectric standard because its thermoelectric power is fairly insensitive to impurities (Pearson 1957) in contrast to some metals like copper (MacDonald and Pearson 1953). Also lead is readily available commercially and can be very readily extruded as a wire.

Absolute Thermoelectric Power of the Specimen Addenda

In order to obtain the absolute thermoelectric power of sodium, it was necessary to know the contribution of the specimen holder and potential leads to the measured thermoelectric power. To determine this a piece of lead wire was mounted in the specimen holder and the resultant thermoelectric power was measured. Using the values for the absolute thermoelectric power of lead (Christian et al. 1958) the absolute thermoelectric power of the specimen addenda was deduced.

Two lead specimens were prepared. The first of these (Pb-1) was of unknown purity obtained in the form of lead foil from the Metallurgy Department. The foil was melted in a test tube and a piece from the center

of the resulting ingot was extruded to make a specimen 5.2 cm long and 0.118 cm in diameter. This specimen was mounted in a copper and inconel specimen holder. The second sample (Pb-2) was made from Tadanac brand[#] intermediate grade lead (purity 99.999%) using the extruder described in appendix II. The method of mounting and the specimen holder used were the same as those used for the sodium and are described in section 3.11. Since the specimen addenda for (Pb-2) was the same as for the two sodium samples the data obtained from (Pb-2) are given in Table II and shown in figure 21.

TABLE II

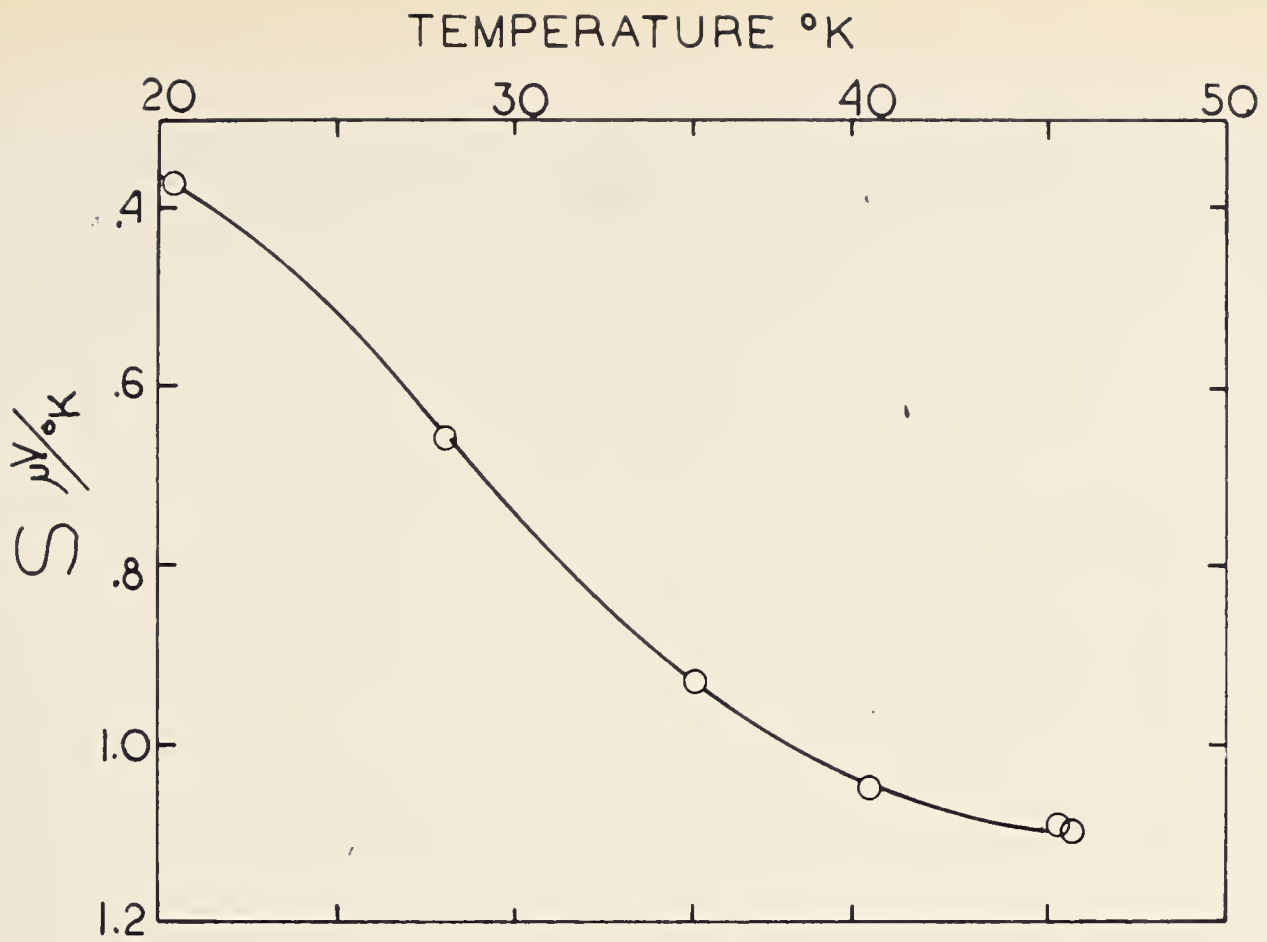
Temperature	Thermoelectric power (specimen addenda & Pb-2)	Absolute Thermoelectric of Specimen Addenda
deg K	$\mu\text{v}/\text{deg}$	$\mu\text{v}/\text{deg}$
9.8	-0.69	-0.27
11.2	-0.77	-0.23
14.9	-1.05	-0.30
20.3	-1.16	-0.37
27.9	-1.43	-0.66
33.0	-1.69	-0.93
39.8	-1.82	-1.05
45.2	-1.86	-1.09
45.6	-1.87	-1.10

Thermal Conductivity and Electrical Resistivity of Lead

The thermal conductivity of (Pb-1) and both the thermal conductivity and electrical resistivity of (Pb-2) were measured.

In pure metals the transport of heat is due almost entirely to conduction electrons and the lattice thermal

[#] We are grateful to Mr. G. H. Turner of Consolidated Mining and Smelting Co. of Canada Ltd, Trail, B. C. who provided this lead.



THERMOELECTRIC POWER OF SPECIMEN ADDENDA

Figure 21.

conductivity may be neglected. It is usual to define the thermal resistivity, $w = 1/k$, where k is the thermal conductivity. Then for a pure metal

$$w = w_0 + w_1 ,$$

where $w_0 = \frac{A}{T}$

and $w_1 = BT^n$ with $n \approx 2$ for $T \leq \theta/10$.

w_0 is the thermal resistivity due to scattering of the electrons by impurities and w_1 is the thermal resistivity due to scattering by lattice vibrations. A and B can then be determined from experimental measurements by plotting wT against T^3 .

The electrical resistivity of a pure metal arises from the same two scattering mechanisms and according to Matthiessen's rule may be written as:

$$\rho = \rho_0 + \rho_i(T) .$$

The residual resistivity, ρ_0 , is due to impurity scattering and ρ_i , the ideal resistivity, to scattering by lattice vibrations. In general the residual resistivity is independent of temperature while the ideal resistivity varies as T^m where $m \approx 5$ for $T \ll \theta$ and $m \approx 1$ for $T > \theta$.

The electrical and thermal conductivities are related by the Lorenz ratio $\frac{k\rho}{T}$, which in the range where the

residual resistance is dominant becomes $L_0 = \frac{\rho_0}{w_0 T} = \frac{\rho_0}{A} ,$

where L_0 is the Lorenz number, 2.45×10^{-8} watt ohm deg⁻².

Since lead becomes superconducting at 7.2°K it is impossible to obtain its residual resistivity by direct measurement. Van den Berg (1948) determined the residual resistivity by applying a magnetic field to destroy superconductivity. In the present work however the residual resistivity of lead was calculated from the thermal conductivity assuming that the Lorenz ratio holds.

Values obtained for the thermal conductivity are given in Table III and it was found that below 10°K the thermal conductivity of (Pb-2) is

$$1/k = W = \frac{0.085}{T} + 330 \times 10^{-5} T^2 \text{ cm deg watt}^{-1},$$

which is in good agreement with the expression given by Mendelssohn and Rosenberg (1952).

<u>TABLE III</u>			
(Pb-1)		(Pb-2)	
Temperature deg K	k watt/cm deg	Temperature deg K	k watt/cm deg
2.31	1.14	2.17	1.39
3.28	1.24	2.64	2.78
3.78	1.38	3.04	2.97
4.27	1.54	3.84	2.58
5.27	2.00	4.8	2.76
6.27	2.56	5.9	3.30
7.05	3.08	6.5	3.65
7.36	3.14	7.35	4.27
7.69	2.82	7.6	3.82
9.68	1.80	9.8	1.92

Values of k above 10°K are not given because our primary aim was to obtain the thermoelectric power of the specimen addenda, thus the copper leads attached to the specimen

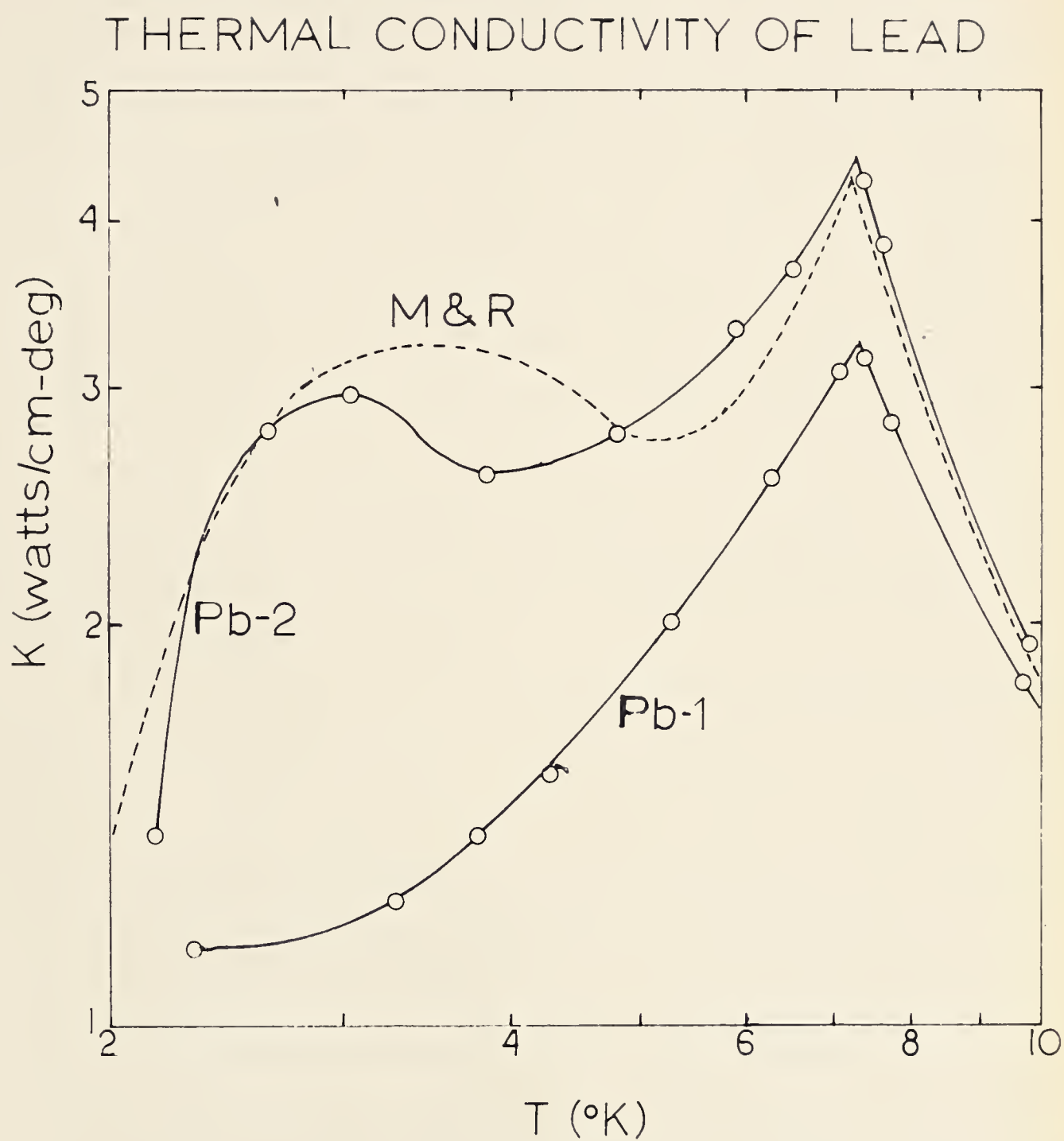


Figure 22.

were anchored at 4°K , providing an undesired heat leak. These leads were made of electrolytic tough pitch copper which has a very high thermal conductivity (Powell, Roder and Rogers 1957) that increases with temperature until it reaches a maximum at about 30°K but remains very high even at 50°K . So even although the copper wires have a very small diameter compared to the specimen they would allow an appreciable heat loss above 10°K , which made the thermal conductivity appear much higher than it really is.

The electrical resistivity of (Pb-2) was also measured. The temperature independent residual resistivity as deduced from the thermal conductivity data, is $0.0021 \mu \text{ ohm cm}$.

The measured resistances had to be corrected to allow for the resistance of the stainless steel part of the specimen holder which was in parallel with the lead. The residual resistivity of stainless steel (or for that matter all except very dilute alloys) is completely dominant below about 50°K , and at higher temperatures, where it becomes appreciable, the ideal resistivity varies linearly with temperature. Using Matthiessen's rule the resistance of the empty specimen holder could thus be deduced at any temperature from the measurements we made on it at 80°K and 300°K . Although Matthiessen's rule may give resistances in error by a few percent for

such an alloy, its accuracy is sufficient for calculation of the small corrections that it was necessary to apply to our readings. The values obtained for the ideal resistivity of lead agree well with those of van den Berg (1948) and are tabulated below.

TABLE IV

Ideal Resistivity of Pb-2

Temperature	Ideal resistivity
°K	μ ohm cm
7.3	0.0141
7.3	0.0140
9.1	0.0828
10.5	0.158
14.5	0.225
19.3	0.549
26.8	1.08
33.9	1.60
38.6	1.98
43.8	2.36

Ideal Resistivity of Lead

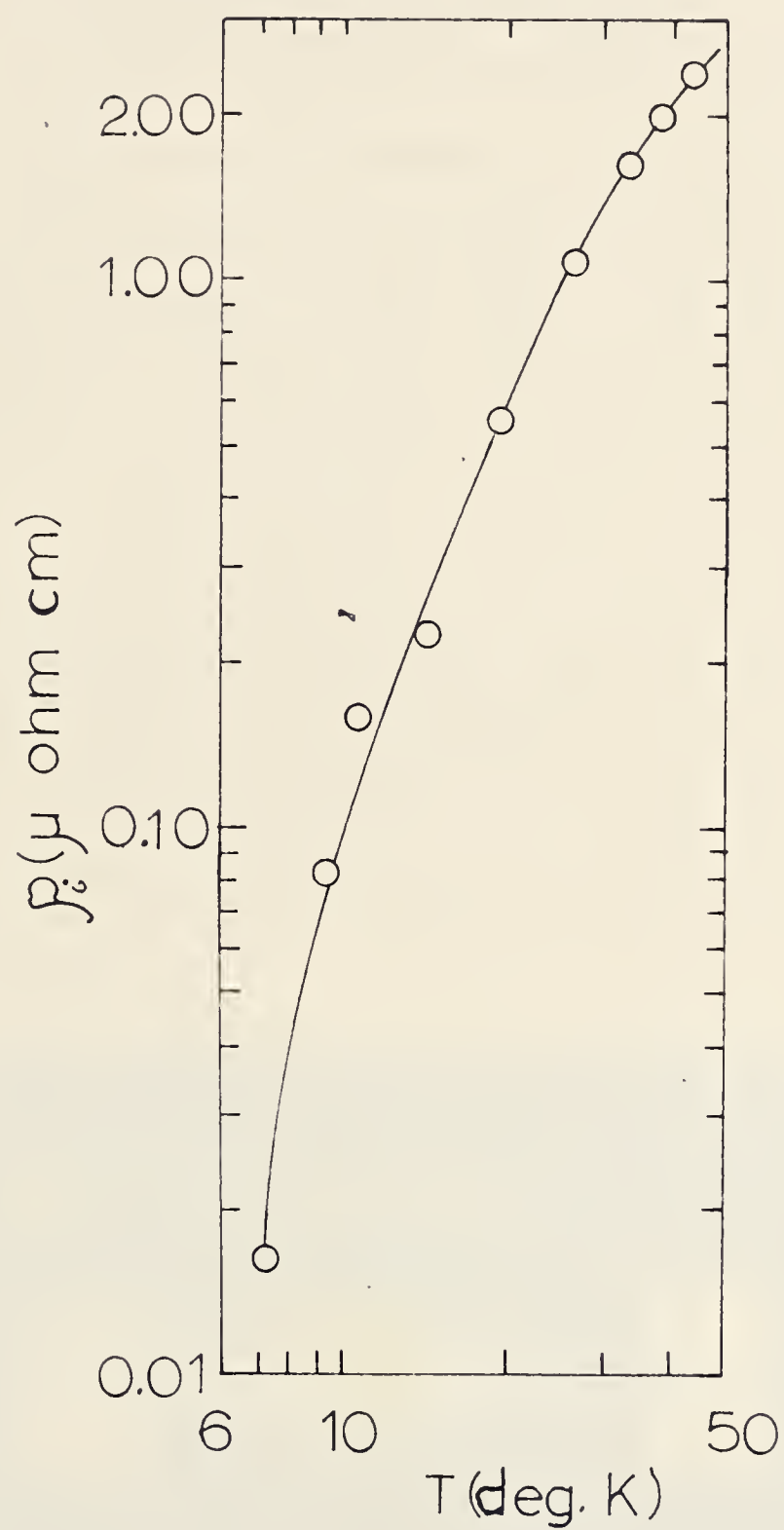


Figure 23.

APPENDIX 2AN EXTRUDER FOR SOFT METALS

In order to produce specimens in the form of wires an extruder was constructed and is shown in figure 24. The whole assembly was made of tool steel. The extruder could be held either in a vise or by a pipe wrench, while the piston was moved by turning the $\frac{1}{4}$ inch bolt at one end of the extruder. A ball joint between the bolt and the extruder enabled the bolt to be turned without turning the piston. A replaceable thin brass washer was placed below the piston to prevent the metal from extruding upward between the piston and cylinder wall. Both lead and sodium were easily extruded using only a six inch wrench to turn the bolt.



Figure 24. An Extruder for Soft Metals

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